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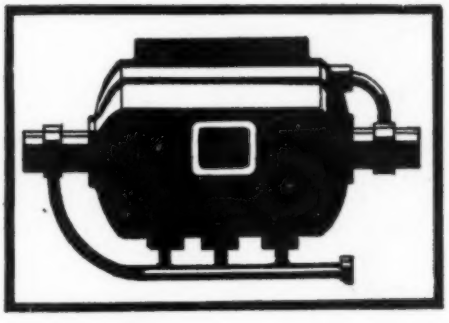

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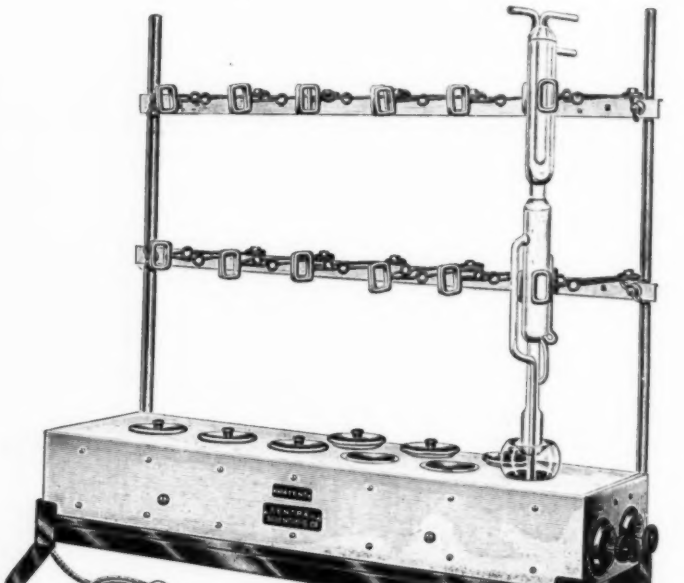

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The Application of Modern Analytical Methods to Transactions in Cottonseed Products

By DAVID WESSON

CONSTANTLY improving analytical methods have done much to remove trading transactions in Cottonseed Products from the realms of rule of thumb, to the basis of more or less scientific accuracy.

The transactions of the American Oil Chemists Society and the work of the Smalley Foundation bear testimony to the constant striving for improvement, which has been going on for many years.

The work of the Smalley Foundation in improving the methods for the analysis of meal, and especially in the determinations of oil and ammonia, have placed transactions, based on this analysis, on a very secure foundation, where the only reasonable chance for error is that of sampling.

The time seems now ripe for a great reform in the commercial practice of selling cottonseed cake and meal.

Other things being equal, the value of these products depends almost entirely on the amount of ammonia or protein present. No producer of metal-containing ores would think of selling the same on any other basis than on the value of the metal contained. Yet thousands of transactions occur where cottonseed meal, often several times as valuable as ores, is simply sold under designations, such as Prime, Choice or Off, without taking into full consideration the true intrinsic values. It would be far better, both for buyer and seller if rules were established whereby cottonseed meal, which is the most highly concentrated protein-bearing cattle food on the market, should be dealt in only on a protein basis, and paid for in accordance with the amount of protein content.

David Wesson

Crude Oil

For years Crude Oil has been handled under trading rules, recognizing the quality of the refined oil produced from it. Its value also depends on the amount of refined oil which the crude oil contains. Our present trading methods depend on an empirical laboratory test which does not tell the actual amount of refined oil con-

tained, but only the amount which a chemist is able to get from a given sample of crude under specific laboratory conditions.

In most highly developed industries dealing with developed materials, it has been customary for many years to value the materials dealt in by the amount of the valuable constituents contained as in the case of metalliferous ores referred to above. In the case of Crude Cottonseed Oil, its value depends on the amount of refined oil which it contains. Our present refining test does not show this. We have available, however, a method which will give within a tenth of a per cent the exact amount of refined oil present in any given sample of crude. By using such a method, discrepancies between different laboratories would disappear, assuming analyses were made by properly skilled chemists. The amount of refined oil from a given sample would depend on the skill employed by the refiner. This of course should not enter into the question of crude oil valuation, except the refiner would naturally know what product he might expect from a given crude.

Color of Oils

This is a matter which has excited a great deal of attention and research during the last thirty years, and the end is somewhere off yet. A method which gave good, workable results nearly thirty years ago, consisted in comparing a column of oil with varying lengths of column of a solution of standard bichromate of potassium. This method, while a little rough, was eminently practical and served for the control of a number of refineries. We next started in using Lovibond glasses, which have become as sacred with many people as the laws of the Medes and Persians. As Dr. Priest has recently shown, the Lovibond glasses vary greatly among themselves. It is possible, however, to obtain a few standard glasses of which the error can be determined. By use of a suitable instrument, such as a Dubosq Colorimeter, it is believed that all practical ends would be served by finding out what depth of a given oil would match a standard glass, and reporting the color accordingly. For instance: if we considered 35 Yellow—7.5 Red, Prime Oil in a 5 inch column, and the sample in question in a depth of 4 inches, matched the standard glass we would have an excess of 20 per cent color over Prime. If a 2½ inch column matched the standard glass, we would have twice as much color in the sample as in Prime Oil. We strongly believe that a system of this kind can be worked out which will do away with a great deal of difficulty which we have in obtaining standard glasses and in making measurements which have no real manufacturing importance and are only useful for hair-splitting distinctions in accepting or rejecting oils for trading purposes.

Flavor

In the good old days when we knew nothing about refining oil, except that caustic soda would remove the free acids and the coloring matter from crude oil, and if the crude oil happened to be made from seed that was more or less damaged, the resultant refined oil would taste bad, flavor was a matter of prime importance. In present day conditions, where all oil has to be deodorized in order to use it for edible purposes, the flavor disappears, and all oils are brought to practically the same level. It

would seem, therefore, that the importance and fine distinctions which are attached to flavor are largely obsolete, and should be given consideration only in deciding whether an oil is prime or off when taken into consideration with analytical factors.

It is the experience of most refiners that a strictly Prime crude oil made from sound seed will not show an acidity of more than 2.5 per cent free fatty acid as oleic acid. An occasional sample of crude will be found where some badly damaged seed has been mixed with sound seed, and if the damage to the seed has been caused by heating, the flavor will be so rank and the difficulty of obtaining good colors with caustic soda and bleaching will become so difficult that such an oil is easily graded as off. Such oils, however, are the exception and met with very seldom.

In conclusion, I wish to say that the time seems ripe for us to leave some of the beaten paths, some of which I helped blaze myself, and strike out into the new and better ways which are clearly indicated by scientific investigation and the progress of the industry.

Editor, Oil & Fat Industries:

One wonders why no more definite attitude, in the form of expression of opinion in this journal, has been taken by cottonseed oil chemists towards the proposal published several months ago by Dr. Wesson to substitute a chemical method for the semi-kitchen one now used in analyzing cottonseed oil.

Considering the effort spent in convincing the industry of the value of chemical service in the rational management and handling of cottonseed products, it is so much more regrettable that the present laboratory method of crude oil analysis lends itself to be used as a tool to undermine the position gained by the chemist.

To illustrate: during a conversation with the manager of the refinery department of a well known company, he referred to "buyers' referee" and "sellers' referee," in the sense that results reported by certain referee laboratories may be expected to be favorable to buyer or seller, as the case may be.

Of the eighty-odd analysts reporting on the check meal sample of the Smalley Foundation, it is unusual to meet with any significant discrepancy in the ammonia percentages. The fairly uniform chemical method of the determination accounts for the uniformity of results.

The laboratory refining test is of course based on the principle of small scale approximation of the large scale process. No need to emphasize that in such cases the large scale process usually has the advantage. But we must admit that there is not necessarily always an invariable relation between the processes of the two different scales. Once a uniform chemical basis method is introduced, the relationship between it and refinery practice will be definitely established, and seller, buyer and chemist will be spared the irritation which is a frequent by-product of the use of the present method.

According to Mr. C. E. McLean, with whom I had occasion to discuss the matter lately, a color reading system of oil could be worked out in connection with the proposed chemical method. S. LOMANITZ.

Winterizing Oils

Progress in Handling Methods Attains Reduction of Refrigeration Needed

By FRANCIS M. TURNER

So far as we know today no actual oil is formed of one and only one Glyceride. Stearin is a glyceride, being formed by the union of Stearic Acid with Glycerol. Palmitine, of Palmitic Acid and Glycerol, is another. There are many others such as Olein, Linolin, Linolein, and so forth. All these Glycerides solidify at different temperatures; for example Stearin and Palmitin solidify at relatively high points, Olein, Linolin and Linolein at relatively low points.

Taking the specific instance of Cottonseed Oil, when this oil, consisting principally of a mixture of Palmitin, Olein and Linolin is chilled, the Palmitin falls out, the Olein and Linolin remaining fluid. The Palmitin is separated out and the liquid oil is then known as 'Destearinized' or 'Winter' Oil.

Many of the Vegetable, Animal and Marine Oils when chilled deposit a mass usually crystalline, although in many instances the precipitation is in gelatinous form. The class name 'Stearin' has been given to these deposits.

It is desirable that oils intended for edible use should be clear at temperatures near the freezing point. In the manufacture of Animal Lubricating Oils, for example, Lard Oil intended for Railroad use, it is important that the oil remain liquid at low temperatures, minus 15° C often being specified for Winter Lard Oil.

Fish Oils such as Menhaden, Sardine, Herring and Cod must undergo destearinization before their Employment in the Arts. Partial purification of oils is effected somewhat during destearinization as albuminous substances are less soluble in cold than in hot oils, consequently, improvement can be accomplished in that manner—especially of Menhaden, Herring, Sardine, Cod and other Fish Oils (See English Patent, Buchanan 1905, German Patent, Megemann.)

Francis M. Turner

The flavor of Medicinal Cod Liver Oil is undoubtedly improved by chilling and removal of the 'Stearin' which oil by the way is one of the most difficult of all oils to destearinize commercially.

Various Processes Employed

The origin of the term 'Winterizing' is obvious. This operation was for a long time effected by storing oils in tanks in cellars and other cool places during the Winter, and 'racking off' the supernatant layer of liquid oil. The old name 'Racked Oil' is still often employed to designate 'Winter Oil.'

This slow and cumbrous process soon gave way to quicker and more efficient methods, as it was too costly to hold large quantities of oil for long periods. Artificial refrigeration was introduced and various methods of applying the chilling medium came with this improvement.

In America the general practice was the gradual chilling of oils until they are, as it is called, 'seeded,' preceding separation by means of filtration usually performed by means of filter-presses; although, in the Fish Oil trade separation of the clear oil from the magma was done with conical filter bags suspended from poles in cool rooms, the clear oil dripping through by gravity. Many plants have chilling tanks, in which the oil is cooled, placed in insulated rooms. Others put the oil in large steel tanks submerged in water, the chilled water circulating around. When the chilling is complete the chilled oil passed through filter-presses, separating the clear 'Winter Oil' from the 'Stearine.'

These are the general methods in vogue, although there are many variations. In England many plants are equipped with chilling tanks which are installed in insulated rooms, the tanks being provided with agitators consisting of piping through which the cooling brine is circulated. After the oil is cooled to the correct point it is filter-pressed in the ordinary manner. Another even more expensive and impractical method—the description of which appears in a recent text book—is conducted in a tank provided with a stationary cooling coil. Below this tank is another into which the cooled oil is supposed to drop and from which it is pumped to a filter press, for separation of liquid and solid parts.

Why, you may ask, are these three methods described above incorrect? The great waste of refrigeration is the greatest drawback, no provision being made for removal of the frozen oil from the chilling surface. That congealed oil is almost a non-conductor is a well known fact. Chilling oil in tanks is exceedingly slow, for which reason a very large amount of refrigeration is wasted. In the case of the last two methods the surface of the cooling coils soon becomes so coated with solidified oil that the transmission of the refrigeration is a slow and very costly process.

A few years ago experiments were instituted in an endeavor to find better methods with lower cost of refrigeration and labor. The latter item is quite high, especially in the winterizing of fish oil. A very complete study of the subject resulted in finding that many of the hypotheses which had prevailed were fallacious. One point tenaciously held was that in the process of chilling the oil the deposited 'crystals' should be kept as large as possible in order to obtain the greatest yield. Repeated experiments proved the contrary to be true; i. e., best practice is to have the crystals as small as possible in order to obtain the greatest yield. Again, what becomes of the crystals after they have passed through the pump enroute to the filter-press? Actually they are

very well churned up, as has been determined by actual observation. The chief objection, however, to all these methods, is the tremendous amount of refrigeration wasted. In all processes today everything is 'figured back to the coal pile,' hence the cost of refrigeration is an important factor in the total cost of winterizing.

1.043 tons are the theoretical tons of refrigeration required to chill 1000 gallons Cottonseed Oil from 90° F. to 38° F. Reduced to gallonage, 1 gallon would require 2.086 lbs. of refrigeration.

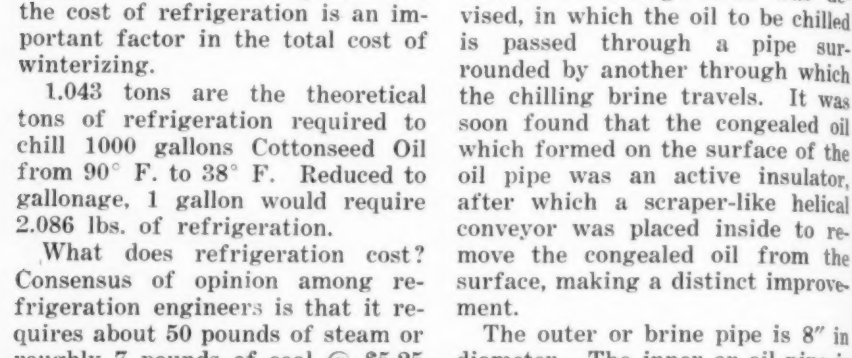
What does refrigeration cost? Consensus of opinion among refrigeration engineers is that it requires about 50 pounds of steam or roughly 7 pounds of coal @ $5.25 per ton to produce 1 ton of refrigeration, therefore the cost is 1.5c per ton refrigeration, approximately.

A New and Improved Method

Permit me to apply these facts to another method of Winterizing or Destearinizing Oil which is now in successful operation in several plants, a method which is superior in efficiency and economy when compared with the methods formerly used. This is the result of a series of experiments conducted with the object of economy of refrigeration and other costs in mind.

The construction of insulated rooms is very costly. Were it possible to apply the refrigeration directly or nearly so to the oil to be treated instead of chilling a room containing a tank of oil an economy could be effected. After many trials an apparatus which would save much refrigeration was devised, in which the oil to be chilled is passed through a pipe surrounded by another through which the chilling brine travels. It was soon found that the congealed oil which formed on the surface of the oil pipe was an active insulator, after which a scraper-like helical conveyor was placed inside to remove the congealed oil from the surface, making a distinct improvement.

The outer or brine pipe is 8″ in diameter. The inner or oil pipe is 6″ in diameter. The helical scraper revolves in the direction of the flow of oil and clears the surface very slightly. The length of the pipes is about 41 feet. Each pipe takes up a space of about 1 square foot; the amount of power required being about $^1/_4$ H. P. for each tube. The minimum capacity of each tube is about 44 gallons per hour which is the operating unit. Larger capacities are obtained by multiplication of these units, usually in pairs.



Continuous Destearinizer

Standard results can be obtained by maintaining the temperature of the chilling brine at the proper differential, for instance, the oil entering the apparatus at 90° F. will pass out at 38° F.

Extraction Experiences in Foreign Lands

By BRUNO HASSEL

THE exceedingly favorable economic conditions which prevailed in the U. S. until recent years have undoubtedly exerted anything but a favorable influence upon economical management in the Oil and Fat Industries. In Europe, on the other hand, and particularly in Germany, the distress caused by the war and post war period has compelled men to devise working methods which, in addition to a full yield, will insure the highest possible quality. Since the great losses and high operating costs of hydraulic pressing plants for oil and fat production could no longer be met, it became necessary to perfect as far as this was possible the extraction process. The tests and experiences extending over many years have brought some very favorable results.

The economic conditions in this country, once the war period and attending conditions over, have become normal again; i.e., supply and demand pretty well balance each other, and the shortage in goods has gradually been followed by large production and stocks of goods. There is no question but that consequent competition has become very keen and that many producers are compelled to keep their production costs at the lowest level. For these reasons the oil and fat producing industry has in an increasing measure paid attention to the extraction system, even though in this country manufacturers are rather sceptical in this respect; for the reason that those extraction systems used here were largely rather defective and frequently led to explosions and conflagrations, and also because the products obtained were not of desirable quality.

As already mentioned, the necessity caused by the war and post-war period has compelled us, particularly in Germany, to bring the extraction system to a certain stage of perfection. The results obtained thereby may be designated as being very good, and have proven the absolute superiority of the extraction over the pressing system. Nobody will expect, of course, that a complete change is to take place from today to tomorrow; but this much is certain, that the Extraction System is slowly

Bruno Hassel

but surely making its way and will gradually displace the pressing system. Prejudice and established customs which always have been an obstacle in the way of progress, because based upon non-experience or one-sided experience only, are also in the way of the development and recognition of the Extraction System in the U. S. These remarks are to contribute to the elimination of both of these factors, for my statements are based upon experiences which I have acquired in many countries.

(2) Nature of the raw materials and of the products obtained.

(3) Cost of installation and of operation.

So far as oil yield is concerned, I only need to call attention to the fact that pressed oil cake always contains 8-10 per cent oil, even though the most modern presses be used. This percentage of oil, however, is in most cases much greater. The extracted materials, however, are degreased to plus or minus 1 per cent. The following figures speak for themselves:

	Press System	Extraction System
Oil yield of the seed	34%	39.4%
Oil yield as relates to oil content	85%	98.5%
Oil loss as related to oil content	15%	1.5%

The oil industry introduced the Extraction System in 1856, and since about 1885 the large plants have used it. Developments were insignificant, however, until 1914, as the systems existing were not adequate. Only after the construction of the first efficient rotating extraction apparatus in 1912 did a great change take place, and aided by the requirements caused by the World War, the extraction problem has been solved technically, principally in Germany.

Extraction by means of solvents makes it possible to work all materials containing oil, fat, resin, wax and paraffin; whereas the use of the pressing system is depending upon the nature of the raw materials, and is therefore limited. In order to make a comparison between production of oil by the press and the extraction system, the following points must be taken into consideration:

(1) Yield in oil.

Rapeseed, with a 40 per cent oil content, has been taken as example: The lower the oil content of the seed, the higher is the percentage of loss. With soya beans, for instance, the loss runs to 45 per cent.

The pressing system requires a far more intensive pre-cleaning and trituration of the oil seed. Some important raw materials such as wool, fuller's earth containing oil, used catalyzers, bones and also damaged oil seeds could not be worked by this system. Oils extracted with benzine are purer and of a much better keeping quality than warm pressed oils. They contain less slime, albumin and coloring matter, are mostly free from resins, therefore lighter and cleaner; also their content of fatty acids is lower, so that their refining is less costly and more easily managed.

The residues from extracted material, which are principally placed on the market as feed stuff, are

more tasty and easier to digest than pressed cake. As feed stuff, they are not inferior to press cake, since the animal organism does not digest more than 20-25 per cent of the oil contained in the press cake; but they do, in view of the lower oil content of the extraction flour, contain more protein, which is much more important in the feeding of animals. Experiences extending over many years in Europe, have proven that extraction flour is much superior to press cake as a feed stuff, and the generally very conservative farmer has also recognized this advantage and now prefers to buy extracted feed stuffs; i.e., such with low oil content. Due to its relatively high oil content, press cake soon gets rancid, since through oxidation fatty acids will form, through which the cake acquires a bad taste, and being acid has an unfavorable influence upon the animal organism. Extracted feed flour can not become rancid, since it contains practically no oil, can be stored for long periods, and is always fresh and tasty. Also, it is in shape to serve as feed, whereas press cake has first to be broken up and ground.

There is also an economy in the cost of the extraction plants, since in the pressing system a greater number of heavy and costly presses, costly power stations, large heat transmission and transport equipment are required, which necessitate considerably more labor, space and heavier foundations than is the case with an extraction plant of the same capacity. In the latter the consumption of steam, power and cooling water, in addition to the labor required and the losses in solvents, are the principal operating costs. How small they are as compared with those in a pressing plant may be seen from the fact that a modern extraction plant of 40-50 tons capacity in 24 hours requires only three operators per shift. The operating costs in a modern extraction plant, for 1000 kilos raw material, consist of 1000-1200 kilos steam consumption, 10-12 cubic meters cooling water consumption, 8-10 H.P. hours power consumption and 8-10 kilos loss of solvents.

Experience has shown us in Europe that oils and fats extracted with benzine are the purest and bring highest prices. Other solvents, such as di- and trichlorethylene have not been able to maintain themselves, since they dissolve practically all matters containing slime, colors, albumin and resin and yield dark oils and fats, containing many impurities. In spite of the absence of danger of fire and explosion in these solvents, benzine has for the reasons named been given preference almost exclusively as a solvent, all the more so since it has been possible to devise a recuperating system which precludes almost completely all danger of fire and explosion. I wish to mention also that in the use of modern extraction methods all oil and residue are completely free from any smell or taste of the solvent employed.

In recent years a great many extraction systems have appeared on the market and it is difficult to draw a line as to the efficiency and economical operation of every one of them. As I have frequently had occasion to test the various systems as to their efficiency and economy, I am probably in a position to give a well rounded out opinion. There are but few specialists in this line who have had the opportunity to

become familiar in a practical way with the various types of extraction plants and the extraction of the very great variety of raw materials. Each will judge on the basis of the experience which he has had with the system he is operating and which he knows how to handle. It is evident from this that there is often a certain prejudice against those systems with which one is not familiar, and one is apt to judge them exactly as those with which one has had bad experiences. No one knows better than I do that the oil producer is perfectly right in having a certain amount of distrust, as many extraction plants are put in which in no way justify the expectations that were promised for them. The few firms who furnish good, modern extraction plants, and which meet all requirements, are hard put to it, since they naturally can not furnish as cheaply as those firms which in spite of great promises turn out a deficient product. And those who know something about the extraction business can only shake their heads when they see what is still being offered today to the oil and fat industry, by certain manufacturers, in the way of antiquated and defective extraction apparatus.

The modern horizontal or vertical extraction plant, with or without agitating device, whose application is limited to the extraction of oil seed, is by far surpassed by the rotary extraction plant. For a number of reasons this system has proven to be the best over a number of years of practical application. The rotary plant in its construction is well thought out, comprehensive, simple and of easy operation. Its advantages as compared with the others consist in smaller steam, cooling water and solvent consumption, though power consumed is about the same. These advantages are due to the fact that in horizontal or vertical apparatus great quantities of direct steam are required to separate the solvent from the residue, whereas in the rotary apparatus the solvent is separated under vacuum while being heated externally. Direct steam is only required for a few minutes in order to eliminate the last traces of the solvent from the residue. This permits the extracted feed flour to come out of the apparatus thoroughly dried, as required by the trade, of light color and with a high protein content; whereas the flour which leaves other apparatus contains at least 25-30 per cent water and has to be dried in special apparatus. Being in contact so long with direct steam, and on account of this subsequent drying process, the protein content is reduced and the color and taste are impaired, so that the flour is inferior to that produced in rotary apparatus. Every specialist in this line knows that material with more than 10 per cent water content is difficult to extract, as it quickly becomes pasty and the oil can not be eliminated completely. Such materials must therefore be submitted to a drying process, and in connection with horizontal or vertical extraction apparatus there is required for this a separate drying plant; whereas the rotary plant is so constructed that drying, extraction and subsequent drying are all attended to in the plant in one operation. The use of vacuum and jacket steam and the continuous movement of the material prevent all overheating, and the feed flour obtained is of proper color and a good taste. Another advantage is that the solvent mixture can be

drawn from the extractor under vacuum; whereas in the vertical plants special pumps are required for this purpose. The emptying of the rotary extractor is automatic and requires a few minutes; whereas the agitating apparatus in the other plants eliminates only a part of the residue and the rest must be scraped out by hand. Due to this and the presence of benzine vapors an occasional spark has led to many an explosion. If through any circumstance or other, trouble is caused with a certain batch of material, the solvent can in that event be eliminated completely from the rotary apparatus under all conditions, before it is opened or emptied, but this is more or less impossible in other systems.

As already stated, the field of operation in the horizontal or vertical extraction plants with or without agitating apparatus is very limited, whereas the rotary apparatus handles, in addition to oil seeds, all other materials, such as fish, oil-containing Fuller's earth, slaughter-house and meat refuse, bones, wool, leather refuse, paraffin refuse, etc. It is especially suitable for wet materials, as there is no need to install costly drying plants.

In this enumeration of facts I believe there has been demonstrated the superiority of the rotary extraction system over the other systems. There are, of course, various types of rotary systems.

The purpose of my dissertation is in the first place to demonstrate the superiority of the extraction over the pressing system; since before long the question will become vital in the United States for the entire industry producing oils and fats as to how to increase production and lower production costs. Every expert is agreed that this can only be done through the extraction system. Another purpose I have had in view is to facilitate for you the choice between the various systems, based on my experience and practical operation extending over many years. In my own experience I have always come back to one type of system, as by its use I was always able to secure the best results.

I have therefore considered it my duty to call your attention to this system by pointing out its great superiority over all other systems, since it unites in itself all the advantages which we expect of a modern extraction plant: reduced consumption of steam, water, power and solvents; increased output of high grade products, free from all smell or taste of the solvent mediums; and is a well thought out, comprehensive construction with limited personnel requirement and elimination of all danger of fire or explosion.

Fundamental Principles of Refining

By C. B. CLUFF
The Procter & Gamble Company

THE following remarks should be understood to apply only to the common alkali refining process, as applied to hydraulic pressed crude cottonseed oil. They are mainly intended to emphasize the more important points brought out in the study of the refining method, made under the direction of the Refining Committee during the past year.

We should first consider the object to be attained. This consists in removing, with the lowest possible loss, those bodies which are not wanted in the product as sold to the ultimate consumer. These are of three general classes: (1) free fatty acids; (2) coloring and resinous matters, together with various other bodies containing phosphorus and nitrogen, all of which are soluble in the oil, or may actually be combined with the glycerides; (3) insoluble matter, such as settlings of meal and moisture.

Generally speaking, the refining procedure consists of two stages:

1. The chemical action between sodium hydroxide and some of those constituents of the crude oil which are to be removed.
2. The conversion of the products formed in the first stage into a form that will separate readily from the refined oil.

We often think of the refining procedure only as a mixing operation, because it is carried out in a mixing apparatus. It is just as important, however, to bear in mind the equally necessary second stage, not a mixing at all, but a separating operation, and this stage should not take on the nature of a mixing operation.

The principal points to be briefly discussed here are:

Choice of lye
Agitation
Temperatures
Separating foots from refined oil
Practical application

Choice of Lye

The sodium hydroxide is necessarily dissolved in water before being applied to the crude oil, for two reasons:

1. To bring it into a condition where it will react chemically.
2. Because water is necessary to dissolve and emulsify the products formed.

The relative amounts of sodium hydroxide and water constitute what we usually speak of as the concentration of the lye. It is found that some oils refine best with strong lye and others with weak lye, even when there is no difference in free fatty acid content. This is at least partly due to the fact that the quantity of impurities present in certain oils requires a lye with more water to form the desired emulsion.

When the F.F.A. in an oil increases, more NaOH is required for neutralization, but the amount of water required does not necessarily increase correspondingly; hence, we generally find stronger lyes best on oils with high F.F.A. On oils of usual average quality, however, it is generally found that lower losses result when relatively weak

lyes are used, as less saponification takes place with such lyes.

It is found in practice that if we use only just enough sodium hydroxide to combine with the acidity as found by the usual test for free fatty acids, we will not remove all of the coloring matters. The presence of a very considerable excess of sodium hydroxide throughout the entire refining is necessary for removing all of the undesired impurities. It is found that if, during the process of refining, the excess sodium hydroxide becomes neutralized by saponification, we do not obtain as light a color in the refined oil at the end of the operation as when an excess of sodium hydroxide remains. While, therefore, a certain excess is necessary in order to obtain proper color, an unnecessarily large excess causes increased saponificaion of neutral oil with a resulting increase in loss. A certain amount of such saponification always takes place unavoidably, and due to this and to other complicating factors, it has never been possible to predict or to calculate exactly the proper quantity or strength of lye to give the best quality of refined oil with the lowest loss. At present, this can only be determined by experimental refinings.

The best we have so far been able to do is to set an upper limit for the amount of sodium hydroxide which may safely be used, and specify certain strengths which have been found to give good results on oils of various F.F.A. content.

It is to be hoped that we may eventually know more about the various constituents of crude oil, so that by some test we may determine the quantity present, just as we now determine the F.F.A., and then calculate from these tests the proper amount and strength of lye to use.

Agitation

The first essential in refining is to cause the sodium hydroxide solution and the crude oil to come into very intimate contact, so that every molecule in the oil comes in contact with free sodium hydroxide. As oil and water do not mix naturally, the desired contact can only be attained by extremely vigorous agitation. This intimate contact is especially difficult to attain on oils with high F.F.A., where the large amount of soap formed tends to hinder or obstruct the action of the caustic on the other unacted-on particles. Even with the most vigorous possibe agitation, the element of time is of importance. It is found that the agitation must be continued for a considerable time in all cases, in order to bring the reaction to anything approaching completeness. On the other hand, if such agitation is continued unnecessarily long, excessive saponification of glycerides takes place, resulting in an increased loss with no improvement in the color. Our work shows that agitation at 250 RPM with a paddle having one-inch blades for a period of five minutes is sufficient in most cases, but on some oils ten minutes gives a noticeably better result. Beyond this point our experiments do not show any improvement, but do show an increased loss. Hence, ten minutes has been adopted as the standard time for all oils.

Temperature

The temperature of mixing is of great importance. The above mentioned agitation for ten minutes is based on a temperature of 18-22°C., or approximately 70°F. Our experiments showed that when mixed

at lower temperatures, down to 50°F., satisfactory results—both as to color and loss—are obtained, but it is not considered practical to require chemists to operate much below ordinary room temperatures where there is no decided advantage to be obtained. Temperatures of 80°F. or above resulted in darker color or higher loss. Hence, 18-22°C. has been adopted as standard. At ordinary temperatures, the F.F.A. react readily, and apparently the resinous and coloring matters are likewise attacked easily, while the glycerides are not saponified to any great extent. At higher temperatures, saponification of neutral oil takes place much faster.

Separating Foots

At the end of the mixing operation at 18-22°C., we have a mixture of refined oil and finely divided foots, the latter term comprising the soap, coloring matter, and other impurities, together with excess of lye and some emulsified oil. This mixture in its present state of division and temperature will not settle and separate the foots readily. If the mixture be filtered at this stage, the refined oil will be found to have the desired good color, and to this extent the refining operation has accomplished its purpose. It is only necessary then to separate the foots, for which purpose they must be brought into a condition where they will settle readily, and do so without carrying down a large amount of neutral oil. This is accomplished by heating to such a temperature as will cause the small particles to soften and coalesce into larger and heavier masses, at the same time liberating most of the oil originally enclosed or entrained in the small particles. We then find that the foots settle so that the refined oil can be drawn off, leaving only a minimum quantity of neutral oil in the foots. It is impossible to produce a foots containing no neutral oil. The optimum temperature for softening the foots so they will settle readily varies greatly with different oils, and with the relative amount of water present. Crude oil containing only a relatively small amount of impurities, or oil which is refined with a weak lye, will usually make a foots which will soften at a relatively low temperature, and vice versa. The minimum temperature can only be determined by experiment in each case. It has been found, however, that temperatures higher than about 60 or 65°C. frequently give trouble by causing the foots to rise to the top, due apparently to the expansion of air bubbles contained in same, so we have set a maximum limit of 60°C. or about 140°F. If the foots do not settle promptly when this temperature is reached, they will usually do so after being held for a few minutes at this temperature. One of the most essential points developed by our experiments is, that in heating the mixture to soften the foots, it is very important to heat quickly. The former theory of heating slowly resulted in no advantage whatever, but on the contrary has led to extremely variable results; and generally speaking to poorer results, especially as regards loss. The longer time taken in heating can only result in an increased saponification of neutral oil. Hence, we find by experiment that the light color obtained in the cold mixing is preserved and the refining loss kept at a minimum if we heat the sample to the desired final temperature rapidly. Heating beyond the minimum softening

temperature up to the beforementioned limit does not injure the refining, provided the sample is not mixed vigorously during this operation. Mixing during heating is unnecessary and undesirable, except to the extent that we keep the mass moving just sufficiently to hold a uniform temperature throughout the mass while heating. A speed of 70 RPM with the 1" paddle has been found sufficient. Additional agitation results only in breaking up the particles of foots or preventing them from properly coalescing so as to settle well.

What we are really trying to do at this stage of the refining operation is to separate two constituents of the mixture, and not to mix them.

Practical Application

This may be considered from the standpoints of both laboratory and factory operation.

For laboratory work, the principles described have been embodied in a revision of the official refining method recommended for adoption by the Refining Committee. It will be necessary to obtain new paddles, and to modify existing refining machines to meet the requirements of two speeds with positive drive, and quick heating. Then it will be necessary for chemists to religiously follow instructions to the letter to obtain results equal to those obtained by others.

In applying these principles to factory work, it is a difficult mechanical problem to obtain the same thoroughness of mixing in a given time on such a large scale, or to heat a large mass of oil to a high temperature as quickly as it can be done on a laboratory sample. Hence, while the same principles should apply, it will probably never be possible to get quite as low losses in a large kettle as in the laboratory.

Report of the Chevreul Prize Committee

By DAVID WESSON

THE Chevreul Prizes are being offered for the three best original articles published in OIL & FAT INDUSTRIES during the season 1926 and 1927.

The judges of the article will be selected from members of the American Chemical Society and the American Institute of Chemical Engineers.

The first prize will be $150.00; the second prize $100.00, and the third prize $50.00.

The Contest closes with the July number of the Journal.

The announcement of the winners will be made at the earliest possible moment.

DAVID WESSON, *Chairman.*

A Method for Proteins

By R. M. CHAPMAN

WHENEVER there are a large number of samples to be run in a short time, errors are liable to creep in; particularly unless the calculations are as simple as possible. And mistakes in calculations are extremely difficult to run down and check up. It was largely as a means of guarding against errors in calculation that the method described herewith was developed.

The method in question was evolved some ten years ago and has been in use in this laboratory ever since. We have had very marked success in checking with other chemists and with the check meal samples sent out by the American Oil Chemists Society. While on the subject of the Smalley Foundation, the writer would like to express his appreciation of it—in its work, it is unique. I would be lost wthout the check meal samples.

When first this method was instituted in our laboratories, our work was mostly on feeds. Since that time we have employed it for other classes of materia!; such as meat scrap, tankage, blood, bone and various classes of mixed products.

Solutions

Caustic Soda: 40 pounds of 76° caustic soda are added to 5-6 gallons of water in a 10 gallon metal container. This should be stirred from time to time for about an hour when all the soda should be in solution. If tested at this point while still hot, the Bé. should be about 45-48°. The solution should then settle for several days and when tested should show about 50° Bé. or better; but not over 55°, as it is then troublesome to handle. It may be siphoned off when clear and we have found that long standing tends to aid clarification. A blank should always be run on this solution, as different parts of the same lot of soda have at times been found to give different results. The writer at one time found a considerable amount of ammonia in caustic soda solution. In making up this solution the caustic should be added continuously until all is in; this is in order to keep the solution hot enough to dissolve the whole amount of caustic in a short time. We use flake caustic in 10 pound cans.

Sodium sulphide: 150 grams of sodium sulphide (fused chips 60 per cent) are added to 4 liters of water and well mixed. A blank should be run on this solution along with the caustic soda, using in both cases the amount used in the determination itself. We have found in most cases that the black sediment in the sodium sulphide solution has no effect on the blank.

Standard sulphuric acid: 8cc of concentrated sulphuric acid 1.84 sp. gr. are used for each liter of solution (in our own case 160cc for 20 liters of solution). This is thoroughly mixed (we use an air mixer) for at least one hour. We then fill a 50cc certified burette with this solution and draw off five 10cc portions, allowing the burette to drain at least five minutes between each portion.

Fifty cc of water are added to each portion and the solutions heated to boiling. Twenty cc of a solution of barium chloride contain-

ing 2 grams of the salt are added slowly to each portion and the solutions held at the boiling point for one hour. The solutions are then filtered through previously prepared and weighed Gooch crucibles or through double Munktell No. 2 filter papers. After ignition and weighing there should be exactly .3333 grams of barium sulphate for each portion. This is equivalent to an acid strength of .014 grams per cc or .025 grams of protein per cc, using the value: protein equals Nitrogen x 6.25. In actual practice the sulphuric acid is adjusted to approximately the correct value by titrating with a previously standardized sodium hydroxide solution and adding the required amount of water or strong acid as the case may be. The final standardization is always made by weighing up as barium sulphate.

Barium chloride solution: In standardizing the sulphuric acid we make a fresh barium chloride solution each time. Ten grams of the salt are dissolved in about 90 cc of water and hydrochloric acid added until a precipitate forms; then enough water added to just dissolve the precipitate when the final volume should be about 100cc. This is divided equally among the five portions of acid as mentioned above. The precipitate of barium sulphate formed in the manner described using this barium chloride solution is granular and very easy to filter.

Standard sodium hydroxide: This solution contains .01142 grams when one cc exactly neutralizes one cc of the standard acid. It should be made to do so. We use a carefully checked 50cc pipette (the same is used for all determinations) and pipette 50cc of the standard acid which is then titrated with the alkali using cochineal or methyl red as indicator. Both indicators give the same results as their theoretical change points are at the same hydrogen ion concentration, and while we have always used cochineal we like the methyl red very much. Two or three drops of a methylene blue solution added to each titration using methyl red gives a very striking change point and can be used to advantage.

Cochineal indicator: Twelve grams of the cochineal powder are dissolved in 200 cc of alcohol. 200 cc of water are then added and the mixture filtered. The filtrate is made to one liter.

Methyl red indicator: This solution contains .200 grams per liter and should be made and kept in strongly alcoholic solution in order to prevent crystallizing out of the methyl red. It can be made also by using a very small amount of alkali (about 0.5 cc of the standard alkali) in the solution, when it will keep indefinitely.

Determination

Two and a half grams of the sample are accurately weighed into a 500 cc Kjehldahl flask. About 0.7 grams of mercuric oxide free from nitrogen and a small piece of paraffin are then added. Then not over 7 grams of sodium sulphate and 25 cc concentrated sulphuric acid. The sodium sulphate shortens the time of digestion at least two-thirds. Shake well in order to moisten all the sample and start the digestion at a low heat. Continue at low heat until all the melt has run down the sides of the flask. This should not take more than ten minutes and then the heat is increased and boiling continued

until the solution is colorless. I have not found any material yet which would not go into complete solution in 45 minutes. The heat is then turned off and the flask allowed to cool for about ten minutes. Water is then added until the flask is slightly less than half full, then two or three pieces of mossy zinc and 15 cc of sodium sulphide solution. Then agitate in order to precipitate all of the mercury. Then 50cc of caustic soda solution are allowed to run down the side of the flask which is next connected with the condenser and receiver and well shaken. The flame is lighted and distillation continued until the receiver contains about 200 cc. As all the ammonia is in the first 100 cc this gives a sufficient margin of safety. The receiver is then titrated with standard sodium hydroxide using cochineal or methyl-red and the reading of the burette subtracted from fifty gives the percentage of protein directly.

Notes on the Determination

Before connecting the flask with the distilling apparatus (we use Sargent's although any standard apparatus suffices) a 300 cc Erlenmeyer flask containing 50 cc standard acid (pipetted) is placed at the receiving end of the condenser. After shaking the flask to be distilled, if the liquid in the receiver rises in the condenser and stays there one may be sure that all connections are tight.

50 cc of the standard acid will take care of all feed samples with the exception of very high cottonseed meal in which case 2 grams should be taken and the result multiplied by 5/4. Naturally on all samples whose protein content is known to be above 50 per cent we take 2 grams or, as in the case of dried blood, 1.25 grams. In the latter case the result is multiplied by 2. In cases where we suspect that the protein content will be about 50 per cent we use 2.5 grams and add our indicator to the receiver; then if the color starts to change we add 10 cc more of standard acid and subtract our titration from 60. We have found that if the solution in the receiver becomes alkaline and is left for even a very short time there is a loss of ammonia.

All samples are ground to pass at least 20 mesh before weighing and cottonseed meal or cake, or other high grade material is ground to a very fine powder.

Tap water is used throughout the determination except in making standard solutions and indicators.

There may be some objections to using 50 cc of standard acid for all determinations—principally that it is a waste of standard solutions and that there is no resultant gain in accuracy. However we believe that the time saved in calculations and the minimum chance for error more than offset this objection.

This method only applies to protein where the factor is 6.25 but it could be worked out for the other factors just as well. It saves useless figuring and I might say that one of our largest feed manufacturers has not had a deficiency claim from any of the state feed control stations in 10 years.

I should like to hear from anyone who is interested in this method.

R. M. CHAPMAN

Some Effects of Atmospheric Oxidation of Cod Liver Oil

A gummy residue, rancidity, acid taste, and destruction of Vitamin A result from storage in contact with air

IN order that we might have definite information concerning the results of storing cod liver oil in partially filled containers, a number of flint glass jugs were filled one-quarter full of cod liver oil and stored under a variety of light and temperature conditions. The jugs have never been opened, yet there was sufficient oxygen in the air that they contained to produce easily recognizable oxidized oil. The accompanying photograph clearly shows the gummy oxidized oil attached to the side of the jug. Without doubt, the oil is very rancid and of a decidedly acid taste. The most serious effect of light and air on oil stored under such conditions is the destruction of its vitamin content.

The effect of oxidation in a partially filled glass jug. Note the gummy, oxidized oil on the side of the jug

In our October issue, we referred to a paper, "Effect of Light on the Vitamin A Content of Cod Liver Oils," which contained the results of a study made by the research laboratory. If you will recall, the results of that investigation showed that when cod liver oil is stored in clear glass bottles, exposed to the light, the vitamin A content of the oil is more or less destroyed depending upon the length of storage and the intensity of the light in which the oil is stored.

Drummond and Zilva have also shown that when cod liver oil was stored in partially filled containers, the vitamin content was markedly diminished. If the oil was stored in bottles filled almost to the top, and tightly stoppered, the vitamin potency was preserved for two years.

From time to time, we receive requests to supply our oil in bulk. The above photograph is printed in order that our readers may visualize for themselves the possible consequence of cod liver oil standing in partially filled containers.

Reprinted from Patchworkings, E. L. Patch Co., Boston

February Imports of Olives, Coconut Meat, Coconut Oil and Copra, Peanuts and Peanut Oil, and Edible Olive Oil

Countries	Olives in brine		Coconut meat, dessicated, cut, etc.	
	Gallons	Dollars	Pounds	Dollars
France	24	181	.....	.....
Greece	4,333	2,477	.....	.....
Italy	6,150	1,716	.....	.....
Portugal	1,512	1,324	.....	.....
Spain	482,365	462,924	.....	.....
Canada	2	3	.....	.....
Cuba	6	12	.....	.....
Peru	9	18	.....	.....
Ceylon	.....	.....	443,525	37,310
Hongkong	28	33	.....	.....
Philippine Islands	.....	.....	2,179,794	194,470
Total	494,429	468,688	2,623,319	231,780

Countries	Copra		Coconut oil	
	Pounds	Dollars	Pounds	Dollars
Canada	38,814	1,825	.....	.....
Jamaica	12,600	639	.....	.....
Dominican Republic	.....	.....	180	25
Japan, including Chosen	447,242	20,088	.....	.....
Philippine Islands	22,735,054	1,002,948	16,153,104	1,270,664
British Oceania	1,285,902	62,447	.....	.....
French Oceania	520,958	21,745	.....	.....
Total	25,040,570	1,109,692	16,153,284	1,270,689

PEANUTS

Countries	Shelled		Not shelled		Peanut oil	
	Pounds	Dollars	Pounds	Dollars	Pounds	Dollars
France	.....	.....	.....	.....	57,326	6,392
Germany	54,125	4,328	.....	.....	.....	.....
Netherland	.....	.....	.....	.....	17,661	1,987
Spain	.....	.....	220	26	.....	.....
Canada	.....	.....	.....	.....	87,440	8,894
China	5,446,745	235,476	374,500	14,626	3,449	400
Hongkong	3,652	488	4,400	499	118,250	12,977
Japan, incl. C...	1,057	21	24,700	1,234	.....	.....
Total	5,505,579	240,313	403,820	16,385	284,126	30,650

EDIBLE OLIVE OIL

Countries	Packages weighing less than 40 pounds		Other	
	Pounds	Dollars	Pounds	Dollars
France	76,769	16,286	567,125	121,187
Italy	2,519,477	494,355	739,264	164,580
Spain	106,367	21,866	1,286,147	268,996
United Kingdom	2,372	532	.....	.....
Canada	5,799	655	.....	.....
Syria	275	62	.....	.....
Total	2,711,059	533,756	2,592,536	554,763

Report of Committee on Determination of Free Fatty Acid of Oil in Seed

R. K. BRODIE, *Chairman*

THE present report divides itself into two parts, corresponding to the double task assigned to the committee; namely, the development of a method for determining free fatty acid of oil in seed and investigation of the relation of free fatty acid to quality of oil.

I. Method for Free Fatty Acid

The development of a suitable method has accounted for the main effort of the committee. The aim has been to propose a method which will be simple as to manipulation, give uniform results in different laboratories, and give a result corresponding closely to the fatty acid of oil produced from the same seed by the customary milling processes. The proposed method. which had best be stated in advance of the discussion of the committee's work, is as follows.

"At least 100 grams of the well mixed sample of seed are heated 30-45 minutes at 100-105 Deg. C. and cooled. The meats are then separated by any laboratory huller or mill that will approximate factory conditions and ground sufficiently to pass 1½ m.m. sieve. Not less than 10 grams of the thoroughly mixed meats are extracted by cold percolation with gasoline boiling below 70 Deg. and the gasoline evaporated off and the oil weighed. 30 c. c. of neutralized denatured alcohol are added and the free fatty acid of the oil is titrated with a standard caustic using alkali blue as an indicator. The free fatty acid is calculated by the formula:

$$\% \text{ F. F. A. equals } \frac{28.2 \times \text{normality of alkali} \times \text{c.c. used}}{\text{Weight of Oil}}$$

Notes: The gasoline percolation should be continued sufficient time to give at least 2 grams of oil.

The addition of a small amount of gasoline to the flask after the alcohol has been added before titrating makes the end point sharper.

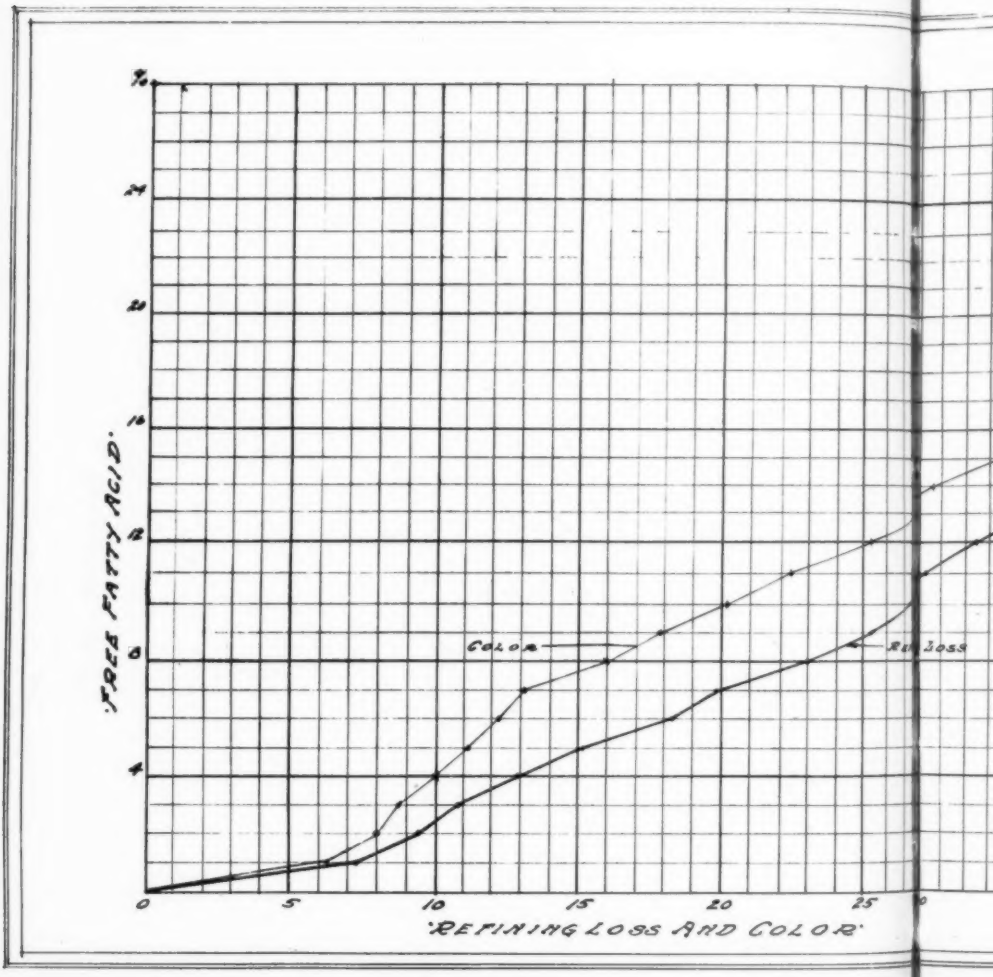
Tenth normal caustic soda is preferable for low fatty acid oils but for oils above 5 per cent, quarter or fifth normal is preferable.

In case it is desired to make the determination where analytical balance is not available, extract a larger quantity of meats and after evaporating all the gasoline from the oil, pipette 7.05 grams and titrate with quarter normal caustic. The reading in this case is percentage directly."

Sampling: The problem of uniform sampling of large bodies of cottonseed is in no way peculiar to the fatty acid determination. Consequently the committee has not concerned itself with the broader aspects of this difficult problem, but study was made of the minimum size of seed sample required for a uniform free fatty acid determination. When prime seed of low fatty acid and off seed of high fatty acid were well mixed in equal quantity, no distinction in the uniformity of observed free fatty acid was noted in samples of 70 grams as compared with larger size, while samples of 35 grams and smaller size gave progressively more erratic results. Also experiments on the mixing of seed dyed with different colors confirm the

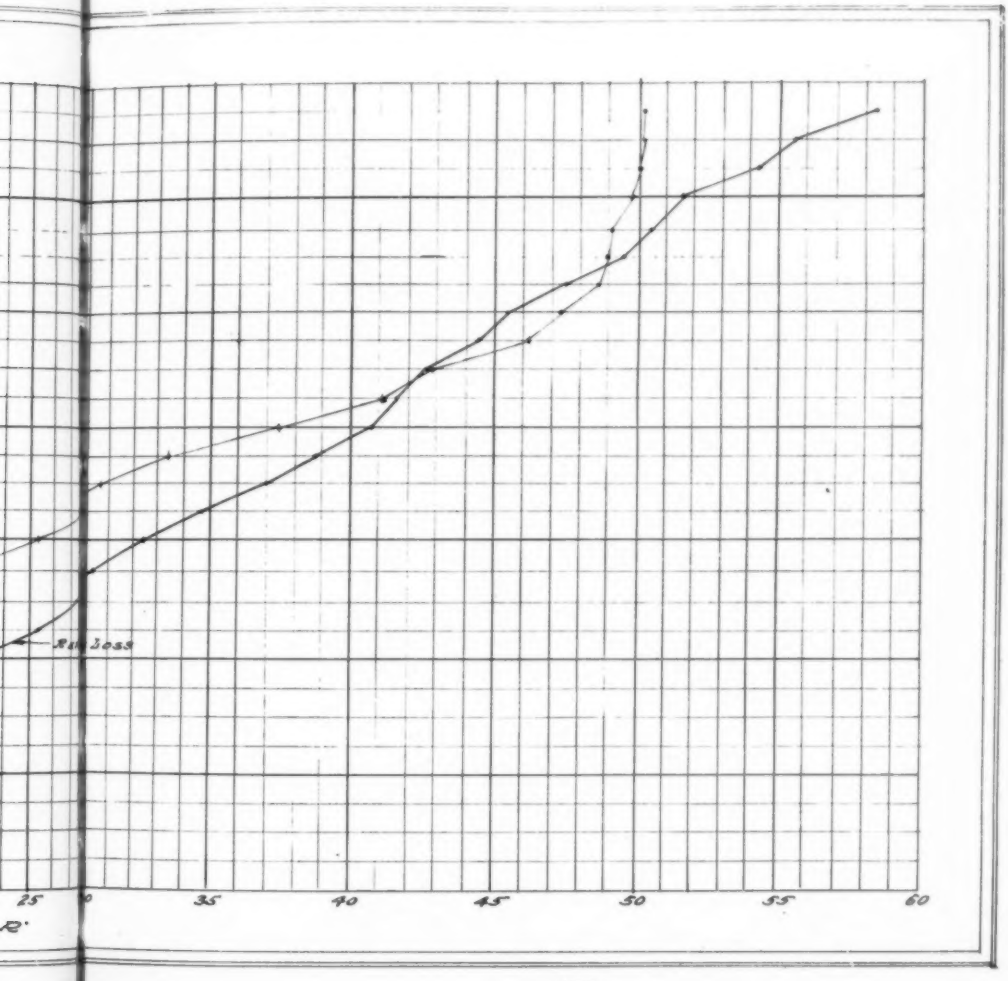
idea that there is a roughly defined minimum size of sample below which increasing lack of uniformity is noted and above which no additional advantage is obtained. The committee believes that a larger sample can be reduced to a representative sample of 100 grams for purpose of determining free fatty acid, provided the larger sample is itself truly representative of the seed in the car or other commercial unit specifically under consideration.

Preliminary Heating of Seed: Under all normal conditions of storage, increase of free fatty acid of oil in whole seed is relatively slow. After hulling, fatty acid in meats increases more rapidly, but still shows no appreciable change within the time ordinarily devoted to an analytical procedure. After rolling or grinding, free fatty acid in meats increases so rapidly that there is danger of results being too high on account of this source of error, especially in seed of high



moisture content. Preliminary heating of seed checks the tendency towards subsequent increase in fatty acid and reduces the chance of error from this source. Although in the committee's own results with the procedure described above no appreciable difference was noted when heating was omitted, the general experience of the committee has been in favor of including this step as an extra precaution.

Crushing the Sample. At the outset it was hoped to devise a very simple procedure involving the crushing of whole seed. After a series of disappointments, the committee reluctantly abandoned even the hope of suggesting an alternative method based on crushing whole seed without separating hulls and meats. It is unnecessary to review this phase of the work, since results were negative, but a few points may be of value in case anyone wishes to undertake further work along this line. Oil ex-



tracted from hulls is higher in free fatty acid than oil from meats, but this factor alone is not very serious. Grinding of whole seed is mechanically difficult and tends to produce a serious rise in free fatty acid which is only partially overcome by preliminary heating. The Copes bag method of crushing, i.e., hammering seed in a canvas bag, does not ordinarily produce increase in free fatty acid, the contrary result being obtained, strangely enough, in some cases. However, this method seems tedious when applied to samples of sufficient size for reliable results: and cannot be recommended.

All factors considered, it seems preferable to separate hulls and meats by any laboratory method which gives a completeness of separation approximating that obtained in ordinary plant practice. The meats can then be ground in almost any type of laboratory mill or household food grinder.

Equipment for Hulling: A number of laboratory mills and even some coffee grinders can be coarsely adjusted so as to serve as hullers. The committee particularly recommends the Bauer Laboratory Grinder, made by The Bauer Bros. Company, Springfield, Ohio. Screening is easily done by hand. A special laboratory machine for hulling and separating, originally made for John Malowan and subsequently modified by The Fort Worth Laboratories, has recently been still further improved and used with considerable success.

Extracting the Oil: This has not been found to be a critical operation. Completeness of extraction and time of extraction are not factors which appreciably affect the results. Simple percolation in the cold is preferred to reflux extraction on account of convenience.

Titration: The committee's experience with the brine method of titrating for free fatty acid was unfavorable. On the average, lower results were obtained by brine titration than by the alcohol method.

Reproducibility: The final results of the committee are frankly disappointing as regards reproducibility. The trouble is believed to be due to the inherent difficulty of sampling. However, reproducibility is good as compared with the methods heretofore employed in seed grading and hence the committee has no apologies to make on this score. Table I gives values for free fatty acid as reported by three different laboratories, working independently on samples from the same lot of prime and off seed respectively and instructed to follow directions given above.

TABLE 1. CHECK DETERMINATIONS OF FREE FATTY ACID OF OIL IN SEED BY THE METHOD RECOMMENDED

Prime Seed		
Lab. 1	Lab. 2	Lab. 3
0.51	0.82	0.9
0.53	0.77	0.7
0.85	0.95	..
0.72	...	..
0.66	...	..
0.82	...	..
0.79	...	..
1.19	...	..

Off Seed		
Lab. 1	Lab. 2	Lab. 3
13.14	12.85	11.3
13.46	13.00	11.9
12.77	12.25	...
13.43	....	...
12.14	....	...
11.08	....	...
12.28	....	...
14.15	....	...

Fatty Acid in Seed vs. Fatty Acid in Expressed Oil

At the outset of the committee's work, there was already available experimental evidence showing good agreement between fatty acid of oil produced by regular milling and oil extracted in the laboratory from the meats used in the mill, provided samples were properly taken and handled thereafter. Subsequent mill experience has confirmed this conclusion. Also in a carefully controlled experiment, a lot of seed which showed 1.5 per cent free fatty acid by the above method of analysis was put through a small experimental oil mill and gave oil of 1.4 per cent free fatty acid. In the case of oil produced on a commercial scale, due to difficulty of sampling, the average agreement is not so good, but is fairly satisfactory. Table II shows the observations made at one mill during thirty-one consecutive days.

TABLE II. RELATION OF FREE FATTY ACID OF OIL IN SEED TO FREE FATTY ACID OF OIL PRODUCED

	Per Cent Free Fatty Acid	
Day	Oil in Seed	Oil Produced
1	2.5	2.5
2	2.8	3.5
3	3.5	3.9
4	3.3	3.6
5	2.4	2.8
6	2.6	3.3
7	1.4	1.4
8	3.9	5.4
9	2.5	1.4
10	4.4	4.4
11	3.8	3.5
12	5.3	5.9
13	5.4	5.0
14	5.7	7.0
15	4.8	3.9
16	6.5	3.9
17	7.3	6.5
18	3.6	4.3
19	4.8	3.6
20	4.1	3.5
21	4.6	4.1
22	6.4	4.0
23	5.0	3.8
24	5.1	4.5
25	4.8	5.3
26	5.1	5.1
27	8.0	6.1
28	5.3	4.9
29	5.5	4.9
30	2.7	2.3
31	2.1	2.1

II. Relation of Free Fatty Acid to Quality of Oil

About 15,000 analyses of crude cottonseed oil from seven independent sources, including both refinery and commercial laboratories, were grouped according to free fatty acid in steps of one per cent (0.5 to 1.5 per cent, 1.6 to 2.5 per cent, etc.) The average free fatty acid, refining loss, and Lovibond red color of the refined oil of the groups are given in Table III. The results are also plotted in the accompanying figure.

TABLE III. RELATION OF FREE FATTY ACID TO QUALITY OF OIL

% F.F.A.	No. Samples	Loss	Color
1	3623	7.25	6.4
2	2662	9.34	8.0
3	1747	10.95	8.8
4	1196	13.00	10.0
5	818	15.06	11.1
6	595	17.21	12.2
7	402	19.96	14.1
8	327	23.00	16.0
9	325	25.36	17.9
10	359	27.50	20.1
11	331	30.35	22.4
12	313	32.48	25.3
13	356	34.70	28.2
14	331	37.00	30.7
15	357	38.80	33.4
16	334	40.80	37.3
17	244	41.72	41.1
18	157	42.82	42.9
19	136	44.52	46.2
20	129	45.42	47.4
21	105	47.45	48.7
22	88	48.62	49.0
23	95	50.40	49.1
24	36	51.60	49.8
25	42	54.20	50.0
26	48	55.50	50.0+
27	49	58.15	50.0+

(*Continued on page* 190)

Report of the Ammonia Committee

Smalley Foundation Cooperative Meal Samples for the 1926-1927 Season

By H. C. MOORE, *Chairman*

IN tables Nos. 1 to 4 following will appear a summary of the results of cooperative analytical work of the Smalley Foundation for oil and ammonia for the past year. This work was concluded with sample No. 30, reported on April 13, 1927. During this year 88 collaborators have participated, as compared to 81, 75 and 78, respectively, for the three preceding years.

Table No. 1 gives the standing of the 43 collaborators who reported oil determinations on all of the samples. Last year only 35 collaborators reported on all of the thirty samples, as compared to 29 and 36, respectively, in the two previous years.

In table No. 2 appears the corresponding standing of the 53 collaborators who reported ammonia results on all of the samples. Last year only 52 collaborators reported on all of the samples, as compared to 42 and 50 in the two preceding years.

Table No. 3 gives the combined laboratory average standing for both oil and ammonia for the 43 collaborators who reported both oil and ammonia on all of the samples. Last year only 34 collaborators reported oil and ammonia results on all 30 samples, as compared to 28 and 36 for the two preceding years.

Table No. 4 gives the summary of the results of other collaborators who have failed to report on all samples, but whose results deserve recognition.

The prize awards for the best work done on the thirty samples are the same as for the past several years, and as published in the

TABLE I—Oil Results, All Samples
(Average analysis, Oil 7.63)

Rank	An. No.	Points off	Av. per sample	Efficiency
1	52	28	.0093	99.878
2	33	34	.0113	99.852
3	57	39	.0130	99.830
4	24	41	.0137	99.818
5	78	45	.0150	99.803
6	45	53	.0177	99.768
7	20	54	.0180	99.764
8	21	55	.0183	99.760
9	23	60	.0200	99.738
10	74	61	.0203	99.734
	73	62	.0207	99.729
12	77	62	.0207	99.729
13	49	74	.0247	99.676
14	22	82	.0273	99.642
15	2	92	.0307	99.598
16	8	99	.0330	99.567
17	4	102	.0340	99.554
18	62	112	.0373	99.511
19	43	117	.0390	99.489
20	42	131	.0437	99.427
21	46	136	.0453	99.406
22	25	140	.0467	99.388
23	71	149	.0497	99.349
24	70	154	.0513	99.328
25	39	167	.0557	99.270
26	37	168	.0560	99.266
27	67	171	.0570	99.253
28	29	178	.0593	99.224
29	6	184	.0613	99.198
30	55	185	.0617	99.191
31	50	194	.0647	99.152
32	3	201	.0670	99.122
33	61	212	.0707	99.073
34	40	257	.0857	98.877
	69	257	.0857	98.877
36	41	285	.0950	98.755
37	76	388	.1293	98.305
38	7	411	.1370	98.204
39	54	446	.1487	98.051
40	82	473	.1577	97.933
41	72	557	.1857	97.566
42	58	608	.2027	97.343
43	63	677	.2257	97.042

TABLE II—Ammonia Results

(Average analysis 8.16)

Rank	An. No.	Points off	Av. per sample	Efficiency
1	31	1	.0003	99.996
2	59	3	.0010	99.988
3	12	6	.0020	99.975
	45	6	.0020	99.975
	10	6	.0020	99.975
6	77	10	.0033	99.960
7	57	11	.0037	99.955
8	41	12	.0040	99.951
9	62	13	.0043	99.947
	33	13	.0043	99.947
11	74	14	.0047	99.942
12	78	15	.0050	99.939
	2	15	.0050	99.939
14	19	18	.0060	99.926
	23	18	.0060	99.926
16	75	19	.0063	99.923
17	73	21	.0070	99.914
18	52	23	.0077	99.906
	55	23	.0077	99.906
	69	23	.0077	99.906
21	24	25	.0083	99.898
	43	25	.0083	99.898
23	42	27	.0090	99.890
24	7	29	.0097	99.881
25	83	34	.0113	99.862
26	25	35	.0117	99.857
27	40	37	.0123	99.849
28	21	38	.0127	99.844
	61	38	.0127	99.844
30	8	39	.0130	99.841
31	72	40	.0133	99.837
32	67	41	.0137	99.832
33	32	42	.0140	99.828
34	27	43	.0143	99.825
35	37	45	.0150	99.816
36	20	49	.0163	99.800
	44	49	.0163	99.800
38	4	52	.0173	99.788
	49	52	.0173	99.788
	76	52	.0173	99.788
41	11	53	.0177	99.783
42	39	62	.0207	99.746
43	16	67	.0223	99.727
44	54	69	.0230	99.718
45	3	75	.0250	99.694
46	6	80	.0267	99.673
47	50	84	.0280	99.657
48	22	85	.0283	99.653
	66	85	.0283	99.653
50	70	86	.0287	99.648
51	17	87	.0290	99.645
52	26	88	.0293	99.641
53	82	106	.0353	99.567
54	64	118	.0393	99.518
55	29	123	.0410	99.498
56	51	126	.0420	99.485
57	71	127	.0423	99.482
58	34	129	.0430	99.470
59	46	130	.0433	99.469
60	36	134	.0447	99.452
61	58	135	.0450	99.449
62	65	140	.0467	99.428
63	30	168	.0560	99.314
64	81	265	.0883	98.918
65	63	277	.0923	98 869

(*Continued in the next column*)

TABLE II—*Continued*

TABLE III—Oil and Ammonia Results, All Samples.

Rank	Analyst	Efficiency
1	33	99.899½
2	57	99.892½
3	52	99.892
4	45	99.871½
5	78	99.871
6	24	99.858
7	77	99.844
8	74	99.838
9	23	99.832
10	73	99.822
11	21	99.802
12	20	99.782
13	2	99.719
14	62	99.729
15	49	99.722
16	8	99.704
17	43	99.694
18	4	99.671
19	42	99.659
20	22	99.648
21	25	99.618
22	6	99.586
23	55	99.549
24	67	99.543
25	37	99.541
26	39	99.508
27	70	99.488
28	61	99.459
29	46	99.437
30	71	99.416
31	7	99.408
32	50	99.405
33	69	99.392
34	40	99.363
35	29	99.361
36	41	99.353
37	76	99.047
38	7	99.042
39	54	98.885
40	82	98.750
41	72	98.702
42	58	98.396
43	63	97.956

Cotton Oil Press in 1923. The winners of these awards for the past year are as follows:

The laboratory cup for the highest efficiency in the determination of both oil and ammonia is awarded to No. 33, Dr. W. F. Hand, State Chemist, A & M College, Mississippi, whose average is 99.899½ per cent. The certificate for second place is awarded to No. 57, A. W. Horrell, Jackson, Mississippi, whose average efficiency is 99.892½ per cent. The corresponding percentages for last year were 99.901 and 99.869, respectively, and for the previous year 99.895 and 99.892.

It should be noted here again that the laboratory cup originally provided for this purpose was awarded finally to the Battle Laboratory, Montgomery, Alabama, having become their permanent property after being won on three different occasions. The cup awarded this year, to be retained by Dr. Hand for one year, is the one which has been provided and offered to the Smalley Foundation by Dr. H. B. Battle.

The certificate for the highest efficiency in the determination of oil is awarded to No. 52, George W. Gooch Laboratories, Los Angeles, California, whose average is 99.878 per cent, and the certificate for second place is awarded to No. 33, Dr. W. F. Hand, State Chemist, A & M College, Mississippi, whose average is 99.852 per cent. The corresponding percentages for last year were 99.871 and 99.786 respectively and for the preceding year 99.880 and 99.848.

The certificate for the highest efficiency in the determination of ammonia is awarded to No. 31, Dr. E. M. Bailey, State Chemist, New Haven, Connecticut, whose average is 99.996 per cent, and the certificate for second place is awarded to No. 59, F. B. Carpenter, Virginia-Carolina Chemical Corporation, Richmond, Virginia, whose average is 99.988 per cent. The corresponding percentages for last year were 99.966 for both first and second place, and for the previous year, 99.956 and 99.942.

TABLE IV—Results of Other Collaborators Whose Results Deserve Recognition.

Analyst	No. samples reported on		Points off Oil	Amm.
5	29	(28S)	192	65
9	28	(27S)	134	39
14	29			38
15	28			85
16	29			152
18	29			116
28	23			182
35	29		639	171
38	29		98	61
68	29		214	433
80	23			61
81	26		428	*
84	29		341	93
85	25		235	12

* 30 samples; reported in Table 2.

In accordance with the resolution adopted by the American Oil Chemists' Society, the identity of the other collaborators will not be disclosed.

It will be observed from the foregoing that the percentage efficiency for the oil work and also for the ammonia work is slightly higher than for last year, although the highest average for both oil and ammonia is slightly under last year's result.

Special attention is called to the very high efficiency in ammonia for No. 31, Dr. E. M. Bailey. Dr. Bailey's record is one which will probably not soon be surpassed. Considering that a variation of two points, that is, .02 per cent is allowed each collaborator on his

Smalley Foundation Prize Winners

A. W. Horrell *F. B. Carpenter*

results as compared with the accepted average on each sample, on only one of the thirty samples did Dr. Bailey's results differ from the average. On the first sample reported his results missed the average three points (.03 per cent). His record is the highest so far established. The record of F. B. Carpenter is not far behind, likewise the records of analysts 12, 45 and 10. The efficiencies of these four collaborators are higher than those of the highest in either of the two preceding years.

The method for determining the standing of the various collaborators and their per cent efficiency is the same as has been used before, and is fully described in the January, 1923, issue of the *Cotton Oil Press*, VI, No. 9, Page 33.

It is hoped that there is no mistake in the results appearing in tables 1 to 4 inclusive. These have been double checked, and yet as there are so many figures involved, there may be some slight error.

The Chairman wishes to thank all the collaborators for their hearty cooperation in this work during the past year. There have been very few complaints on the samples, probably fewer than in former years, and the Chairman feels that a vote of thanks and appreciation is due R. F. Monsalvatge for his painstaking care in the preparation and handling of the samples. The Chairman further wishes to take this opportunity of recommending that this important work be again entrusted to Mr. Monsalvatge next year.

By way of confirming the Chairman's opinion that either the samples during the past year have been more uniform than heretofore

The Mississippi State Chemical Laboratory, another prize winner

or else the collaborators' work in general is improving, attention is called to the following:

Last year the average of all the accepted results for oil was 5.97 per cent; this year 7.63 per cent. The average number of points off in oil for the ten highest last year was 49.9; this year 46.9. The average efficiency in oil for the ten highest last year was 99.721; this year 99.795.

Likewise for the ammonia results, the average of the accepted values for last year was 6.79 per cent; this year 8.16 per cent. The average number of points off for the ten highest last year was 12.5; this year 8.1. The average efficiency for the ten highest last year was 99.939; this year 99.967.

It will therefore be noted that the average of both the oil and ammonia content of the samples this year is higher than last year, yet the average of the number of points off is less and the efficiency higher. It will be noted from an examination of tables 1 and 2 in the report for this year, as compared to the report for last year, that not only is this true for the ten highest, but it is true for the entire list. This would seem to be good evidence of even greater uniformity in the samples this year than last.

An opportunity has been afforded all collaborators to be advised by wire collect, in case their reports are not received in time each week, or in case there seems to be a typographical error in their reports. Thirty-two of the collaborators have taken advantage of this offer, while one or two of the others have been disappointed in finding their results omitted from some report. According to our rule, only results which are received up to and including Tuesday of each week are to be accepted; however

(*Continued on page* 204)

Report of Committee to Develop a Method for Color Comparison of Cottonseed Meal

By R. K. BRODIE, *Chairman*

AFTER numerous attempts to compare cottonseed meal with the color card, the method described as "Method II," below, was decided upon as most satisfactory for comparison, *in situ*.

Due to particles of hull present in the meal, it was the general opinion that a method of rotating the meal and color standard should prove more satisfactory. With this in mind, a rotation disc was prepared with a central depression for meal, the color disc and meal both being covered with a thin sheet of glass.

The disc was found unsatisfactory for three reasons:

a. Difficulty in obtaining a balanced outfit for speeds of 2000 R.P.M.

b. Changing meal samples was slow and unsatisfactory.

c. Too much reflection from the glass cover.

The cup method described as Method I is not as satisfactory as the disc from the standpoint of having only a small surface for observation; but it is easily constructed, balanced, and can be readily cleaned. We believe the rotating cup method will be necessary for relatively few samples, and may even be dispensed with entirely if the color standard can be made to resemble meal more closely.

The Committee realizes that color comparison with the standard is not always readily carried out, due to failure to match in chrome, etc., and that this comparison can be made easier by fine grinding of the meal. On the other hand, very fine grinding in the laboratory for grading purposes would seem to favor poor grinding by the producer. For this reason, the Committee has specified the minimum fineness for cake samples which will give reasonably concordant results.

R. K. Brodie

Color Standards: As described in Interstate Cottonseed Crushers' Association Rule No. 102.

Method of Comparison:

All comparisons should be made upon a neutral gray background. Gray photographic mounting board is convenient for this purpose.

Method I.

a. Meal

This involvs the use of a rotat-

ing comparator of the following description:

"The instrument consists of a two-inch section of two-inch heavy walled clear glass tubing (Pyrex recommended, but oil cup glass will do), mounted on a vertical motor driven shaft which can be rotated at 2000 to 3000 R.P.M. The whole instrument is painted a neutral gray, and is mounted in front of a gray background in good daylight, free from shadows."

The glass cylinder should be half filled with the meal to be graded, and leveled off in order that good balance will be obtained when the cylinder is rotated. A strip of the color standard, seven inches by one inch, should be fitted inside the cylinder above the meal, color side out. It should lie in close contact with the glass; the inner surface of the cylinder being completely covered by the meal and the standard. The cylinder should now be rotated and observed from a position about five feet distant, and not more than one foot above or below the level of the cylinder, a position chosen which will give as much freedom from high-lights and shadows as possible.

b. *Cake*

A representative portion of the cake to be graded should be ground so that 85 per cent will pass a 20-mesh screen, and 75 per cent thru 30-mesh. Portions of sample used for screen test should not be used for color comparison. The ground sample should be graded as for meal.

Note: Any samples of meal containing coarse particles should be ground to the standard for cake, and this fact should be stated in the report.

Method II.

a. *Meal*

The meal to be graded should be placed in the center of a gray sheet or board at least eight inches squared; it should be flattened out to make a level circle about three or four inches across, and a clean, one-inch square of the color standard laid on the center of the meal. The meal and standard, lying in a horizontal plane, should then be observed, in good daylight, from a position directly above them and at least 36 inches distant. For making close decisions, it is best to lay the board on the floor and observe it from a standing position directly above. To be graded "prime," the meal must be as light or a lighter shade than the standard. If darker, it must be graded "off" in color.

b. *Cake*

Grind cake as for Method I, and grade as for meal, above.

R. K. BRODIE, *Chairman*,
T. C. LAW,
H. C. HAMNER,
J. R. MAYS, JR.

Report of the Neutral Oil Committee

By CLAUDE E. McLEAN, *Chairman*

THE work of this committee was undertaken to try to establish a definite relationship, if any, between the refining loss obtained by the present official method and the percentage of neutral oil in the sample. The method used for determining the neutral oil is the one worked out several years ago by Dr. David Wesson with certain changes in the procedure suggested by Dr. George S. Jamieson.

Only a limited amount of work has been done this year. The results of the analyses are shown in the table. In the first column is shown the percentage of free fatty acids determined in the usual way. The second column gives the refining loss by the official method, that given being the best result, considered with color also, of from two to four refinings. The color, with Lovibond glasses, is given in the third column. While the neutral oil results of the three members of the committee are given in columns four, five and six.

It will be noted that Dr. Jamieson's analyses show remarkably close agreement, while those of the chairman are very erratic. This is explained by lack of familiarity with the technique of the method. Our experience so far is that a large number of tests are necessary in order to acquire the technique and also that directions given in the method must be followed exactly. Unless this is done one is likely to become discouraged and think the method unreliable. It is suggested also that until one is thoroughly familiar with the technique it will perhaps be much better to work only on thoroughly settled or filtered samples of oil in order to be sure that the several portions weighed will be identical. We did not do that in this work but took the regular tank car shipping samples just as they came to the laboratory. Practically every sample contained an unusually large amount of settlings, which may have had considerable influence in checking.

Claude E. McLean

The losses by the neutral oil method are uniformly from 45 per cent to 50 per cent of the losses by the best results of several refinings obtained by the official method. It might be of interest also to note that the color of the neutral oil obtained from a mixture of the first four samples was 20 yellow, 2 red while that from the last four, which were of a much poorer quality of crude oil, was 20 yellow, 3 red.

ANALYSES OF CRUDE COTTON SEED OIL

	F. F. A. %	Loss %	Color 35Y and Red	McLean %	Neutral Oil Jamieson %	Putland %
1	1.50	9.40	7.6	95.88		
				95.75		
2	2.90	14.00	7.4	93.12		
				93.02		
3	1.50	8.90	6.7	94.75		
				95.96		
				95.54		
4	1.60	7.60	5.0	96.30		
				96.00		
				96.37		
				96.10		
				97.69 } Same as No. 4, except oil		
				97.08 } thoroughly settled.		
5	3.70	14.30	9.2	92.51		
				91.86		
6	2.95	13.20	7.6	93.47	94.31	93.60
				94.05	94.22	93.60
				93.94	95.94*	94.25
				93.93		
7	3.10	12.20	8.3	94.73	94.60	93.20
				94.45	94.65	92.80
				94.44	94.70	92.90
				94.42		
8	2.00	8.40	5.7	95.35	95.87	93.50
				95.36	95.87	93.70
				94.60		
				95.54		

* Filtered.

Every chemist who has to analyze vegetable oils knows that our present methods of determining the value of such oils are crude and unscientific. Even though the laboratory method simulates the refinery practice it is at best only fairly satisfactory for fixing the value and depends entirely too much on the skill of the operator. We need better and more scientific methods of analysis for the evaluation of these oils. The neutral oil method we feel gives promise of good results and it is recommended that work with the method be continued.

CLAUDE E. MCLEAN, *Chairman.*

Continued from page 181

Acknowledgment

Instead of making personal acknowledgment of the assistance received from many individuals in the above work, the committee members will merely state that the work has been not primarily their own but that of the business organizations with which they are associated. Outside assistance has also been received from the following: The Fort Worth Laboratories; Law & Co.; Portsmouth Cotton Oil Co.; and Seaboard Refining Co.

R. K. BRODIE, *Chairman,*
The Procter & Gamble Company,
Cincinnati, Ohio.
C. H. COX,
Barrow-Agee Laboratories,
Memphis, Tenn.
W. D. HUTCHINS,
The Southern Cotton Oil Company,
Savannah, Ga.

Report of the Detergents Committee of the A. O. C. S., for 1927

By L. F. HOYT, *Chairman*

THE Detergents Committee has devoted its time this past year wholly to the study of methods of evaluating detergents.

I—SCOPE OF COLLABORATIVE WORK

Collaborative work for 1927 has been devoted chiefly to the following options of the determination of detergency.

Option 1. Study of methods for evaluating the color of soiled and washed cloths.

Option 2. Comparative detergent value of eight soaps of widely different composition by substantially the same laboratory washing test as used by collaborators in 1926.

Option 3. Effect of various amounts of added fillers on the detergent value of the same soap, as measured by this same washing test.

Samples and Procedure

Neither of the samples of soap used in 1926 was of satisfactory purity and accordingly for this year's work a bulk supply of a commercial flake soap was obtained and furnished to all collaborators. This soap, hereinafter referred to as "1927 Detergent Committee Soap," was analyzed by standard methods of the A.C.S. with the following results:

Moisture	1.83%
Free Alkali	0.00
Free Fatty Acid	0.00
Unsaponifiable	0.06
Alcohol Insol.	0.042
Water Insol.	0.018
Salt, NaCl	0.88
Glycerin (Acetin)	0.39
Total Alkali, as Na_2O	10.86
(Net anhydrous soap, by difference)	96.80)
Titer of Fatty Acids	35.05°C

The cotton sheeting supplied for the 1927 collaborative work was of improved quality and was Dwight's "Anchor" Brand, Bleached. This sheeting contains no sizing, shows a thread count of 75 x 71 and weighs 4.5 oz. per square yard.

Instead of the separate soiling ingredients supplied in 1926, a paste was made by grinding in a paint mill the ingredients in the proportions previously used; i.e. 50 parts edible tallow, 30 parts lubricating oil (100 per cent Pennsylvania base, 300 seconds viscosity at 100°F) and 20 parts lampblack, by weight.

Directions and Notes

1. Distilled Water is to be used throughout in washing and rinsing.

2. Substitution of the 1927 soiling paste (same ingredients as used in 1926, ground to a uniform paste) is recommended as simpler than the use of the separate ingredients used in 1926.

3. Soiled cloths should not be over 24 hours old at the time they are used for washing tests.

4. In order to reduce the time and the excessive amounts of distilled water required in carrying out last year's tests of 5 washings, each followed by rinsings, the washing procedure has been simplified to give 2 washings each of 15 minutes' duration at a fixed temperature.

5. If time is lacking to make

complete series of tests both at 100°F and 160°F, the tests at 160°F should be done first since it is believed that this temperature more nearly approximates average laundry and washing machine temperatures.

II—PROCEDURE

Option 1. Study of methods of color evaluation of soiled and washed cloths

(A) Soiling: Although the 1927 sheeting contains no sizing, it will soil more easily and uniformly if first washed in distilled or soft water. Rinse, dry and iron flat.

Prepare a solution of 10 grams of 1927 soiling paste in 2 liters of carbon tetrachloride, filtering through a fine screen (i.e. such as 200 mesh) to remove any undispersed particles. Preserve the solution in stoppered bottles and protect from evaporation as much as possible during the soiling operation.

Cut the washed sheeting into convenient lengths, pour a quantity of soiling solution into a dish or tray, pass the cloth back and forth through the solution until it is thoroughly wetted. Then, pass it through a wringer and dry at room temperature.

The object of the soiling operation is to secure a uniformly soiled cloth of a shade equal to S 48 of the Munsell Paper Scale.

(B) Washing Test: Fasten a strip of soiled cloth, S 48, on the wash wheel. Prepare 1 liter of a solution of the soap to be tested containing 5 grams of soap (anhydrous basis) and heat to the prescribed temperature, pour the solution into the wash wheel and run the wheel at 250 r.p.m. for 15 minutes, maintaining the prescribed temperature during that time with the aid of external heat. Stop the machine, drain off the soap solution, add 1 liter of distilled water, run the wash wheel for one minute, drain and repeat the rinsing twice more. Remove the cloth and when nearly dry press with a warm iron. Fold to 4 thicknesses and record the color of the washed cloth in terms of the Munsell Paper Scale. Repeat the washing and rinsing process with the same soap at the same temperature on this same cloth and again

Personnel of the 1926 Detergents Committee

Mr. H. C. Bennett, Los Angeles Soap Co., Los Angeles, Cal.
Mr. J. S. Boulden, Wm. Waltke & Co., St. Louis, Mo.
Mr. V. K. Cassady, Palmolive Co., Milwaukee, Wis.
Mr. A. K. Church, Lever Bros. Co., Cambridge, Mass.
Mr. F. H. Guernsey, Cowles Detergent Co., Lockport, N. Y.
Mr. C. P. Long, Globe Soap Co., St. Bernard, Ohio.
Mr. E. T. Marceau, Goldust Corporation, 44 Beaver St., New York City.
Professor E. B. Millard, Massachusetts Institute of Technology, Cambridge, Mass.
Mr. H. S. Mitchell, Swift & Co., Chicago, Ill.
Dr. W. C. Preston, Procter & Gamble Co., Ivorydale, Ohio.
Mr. W. T. Reese, Peet Bros., Kansas City, Kansas.
Mr. A. S. Richardson, Procter & Gamble Co., Ivorydale, Ohio.
Professor F. H. Rhodes, Cornell University, Ithaca, N. Y.
Mr. F. W. Smither, Bureau of Standards, Washington, D. C.
Mr. T. G. Vail, Philadelphia Quartz Co., Phila., Pa.
Mr. L. F. Hoyt, Larkin Co., Inc., Buffalo, N. Y., *Chairman.*

record the color after the second washing of 15 minutes.

Option 2: Comparative Detergent Value of Different Soaps used under the same conditions.

The Soaps offered for comparison are:

1—Soap Fakes; a commercially pure flake soap free from fillers; moisture content 1.83 per cent when packed. (Note: This soap is the 1927 Detergent Committee Soap).

2—Commercial Soap Powder; composition as packed:—
Moisture 39.4%
Anhydrous Soap 18.1%
Soda Ash 39.8%
Salt 2.8%

3—Commercial Bar Laundry Soap; No rosin; a boiled tallow-coconut oil soap, silicate-filled.
Moisture 38.3%
Anhydrous Soap 45.5%

4—Potash—Coconut Oil Liquid Soap
Moisture 77.97%
Glycerine 2.69%
Total solids 22.04%
Anhydrous, Glycerine-free soap by difference 19.35%

No. 5—8 cold-made Potash Soaps (Approx. 50 per cent Moisture and containing not over 0.05 per cent free alkali (KOH) nor over 0.5 per cent unsaponified saponifiable matter).

5—Potash—Castor Oil Soap
Moisture 48.5%
Glycerine 4.6%

6—Potash—Corn Oil Soap
Moisture 48.0%
Glycerine 4.8%

7—Potash—Linseed Oil Soap
Moisture 48.7%
Glycerine 4.8%

8—Potassium oleate, made from C. P. KOH and Oleic Acid
Moisture 50.6%
Glycerine .....Trace

Washing test to be run on this group of soap products as outlined in the procedure, first at 160°F, and if time permits at 100°F.

The solution used for washing should contain an amount of the sample equivalent to 5 grams of anhydrous sample per liter.

Option 3: Effect of Alkaline Salts on Detergent Action of 1927 Committee Soap.

Conditions of Test:

Soiled cloths to match S-48.

Distilled water to be used throughout.

Concentration of detergent solution to be 5 grams per liter, anhydrous basis.

Temperature of washing, 160°F, 15 minutes; rinse 3 times at 160°F, dry, iron and note color by the Munsell scale. Repeat washing and rinsing process and again note color. (If time permits, washing tests may be made also at 100°F for comparison.)

Note: In determining the weight of soap flakes to use either the moisture content of the flakes should be determined by the collaborator at the time of use or the value of 1.83 per cent (moisture as packed) should be assumed and the weights of soap indicated in the table be multiplied by the corresponding factor which for 1.83 per cent moisture is 1.02, in round numbers.

(A) Soda Ash

Grams 1927 Committee Soap (Dry Basis)	Grams Soda Ash	Per Cent Filler (Dry Basis)
5.0	*0.0	*0.0
4.875	0.125	2.5
4.75	0.25	5
4.5	0.50	10
4.0	1.0	20

(B) Silicate of Soda, N Brand (9.0% Na_2O); 29% SiO_2; 38% solids.)

4.875	0.33	2.5
4.75	0.66	5
4.5	1.32	10
4.0	2.64	20

* Blank.

III—RESULTS OF COLLABORATIVE WORK

Option 1. Study of Color Measurement of Soiled and Washed cloths.

(a) The chairman sent a collection of 12 soiled and washed cloths to nine members of the committee to determine how closely collaborators would agree in matching the color of the same set of cloths with identical color standards. Results are tabluated in Table I and show the personal element enters largely into the use of the present Munsell color scale.

(b) Professor Rhodes of Cornell University contributed generously to the collaborative work of the Detergents Committee by carrying on extensive experiments in the evaluation of the color of soiled and washed cloths by means of a reflectometer (of the type described by A. H. Taylor, Scientific Paper No. 405 (1920) of the Bureau of Standards), equipped with a precision photometer. By means of this device Dr. Rhodes was able to estimate the color of soiled and washed cloths to within 2 per cent and considers the apparatus he used much superior to the Munsell Scale.

(c) Dr. W. C. Preston of the Procter and Gamble Co. continued work begun last season on the measurement of color of soiled and washed cloths by means of the rotating disc method in which a 4 inch sample disc is superimposed on 7 inch slit discs of white and of standard soiled, or black, sheeting so arranged that the proportion of white and of gray or black of the larger discs can be varied at will until the resulting gray, obtained by rotating the discs at a high speed of about 5,000 r.p.m. matches the sample. Dr. Preston finds this method distinctly superior to the present Munsell Color Scale of gray papers for detecting and recording small differences in shade of soiled cloths.

TABLE I

MUNSELL COLOR SCALE READINGS OF TEST CLOTHS

Test Cloth Number

Observer	1	2	3	4	5	6	7	8	9	10	11	12
(1 ft.) Across room.	48	76	67	52	67	64	89+	71	60	64	—48	64
L. F. Hoyt, 1 ft..	48	80	81	52–55	64–67	60–64	89+	71	55	60	—48	60–64
L. F. Hoyt, 15 ft.	—48	76	67	48	64	60	89+	71	60	64	—48	64
F. H. Guernsey, 1 ft.	48	76	71	52	64	64	89+	71	60	60	—48	64
F. H. Guernsey,15 ft.	48	76	71	54	80	67	89+	80	64	67	—48	71
W. C. Preston, 1 ft.	52	89	80	55	67	60	89+	71	55	55–60	—48	60–64
W. C. Preston, 17 ft.	48	80	67	48–52								
C. P. Long					71	64	89+	71	55	60	—48	64
(1 ft.) Along room.	—48	76	67	48	67	60–64	89+	71	55	—60	—48	60
(15 ft.) Across room	48	76	67	52	71	64	89+	71	55	60	—48	64
(15 ft.) Along room.	—48	—76	64–67	—48	64	60	89+	67–71	52–55	55–60	—48	60
H. S. Mitchel, 1 ft.	48	76	64	52	64	60	89+	67	55	60	—48	60
H. S. Mitchel, 15 ft.	52	80	76	55	71	67	89+	71	60	64	—48	67
M. L. Sheely, 1 ft.	—48	80	67	52	67	64	89+	67	55	60	—48	64
M. L. Sheely, 15 ft.	55	83	71	60	67	67	89+	71	64	64	—48	71
J. S. Boulden.												
(Arms length)...	48	80	71	52	71	64	89+	71	55	60	—48	64
Daylight lamp...												
(15 ft.).....	48	80	67	52	71	67	89+	71	60	64	—48	64
W. J. Reese, 1 ft..	—48	80	67	—52	67	64	89+	71	55	60	—48	64
W. J. Reese, 15 ft.	48	71	64	52	67	64	89+	67	55+	64	—48	67
F. W. Smither												
(Arms length)..	—48	76	64	—48	64	55	*83	67	52	55	—48	64
J. H. Bower, 15 ft.	—48	76	64	—48	64	60	*89	67	55	55	—48	60

*Cloth somewhat soiled from handling.

Note: (—) Preceding a number indicates that the cloth was less white or darker than the standard.

(+) Following a number indicates that the cloth was whiter or lighter than the standard.

TABLE II

1927 A. O. C. S. Detergency Test

Option 2

Comparative Detergent Value of Different Soaps Used Under the Same Condition

(J. S. Boulden, Wm. Waltke Co.)

No.	Kind of Soap	Color on Munsell Scale: 1 Wash 160° F.	2 Washes 160° F.	1 Wash 100° F.	2 Washes 100° F.
1	Committee Samples, Commercial Flake Soap...	60+	64	60+	64
2	Commercial Soap Powder..	60	60	—60	60
3	Commercial Bar Laundry Silicate Filled	64	67	55+	60
4	Potash Coconut Oil Liquid Soap	60	64	60	64
5	Potash Castor Oil Soap....	—60	60	55	55
6	Potash Corn Oil Soap.....	60	64	60	64
7	Potash Linseed Oil Soap..	60	60+	60	60+
8	Potassium Oleate	60+	64	60+	64

Readings were made by holding cloth at arm's length under a daylight lamp.

(H. C. Bennett and E. L. Northrup, Los Angeles Soap Co.)

No.	Kind of Soap	Color on Munsell Scale: 1 Wash 160° F.	2 Washes 160° F.	1 Wash 100° F.	2 Washes 100° F.
1	Committee Sample, Flake Soap	67	67	55	60
2	Soap Powder	60	60	52+	52
3	Commercial Bar Soap	60	60+	—55	55
4	K—Coconut	55+	60+	55	60
5	K—Castor	55	55	52	52
6	K—Corn	60	60+	52	—55
7	K—Linseed	60+	64+	52	55
8	K—Oleate	64	64	55	55+

(L. F. Hoyt, Larkin Co. Inc.)

(5 grams, anhydrous basis, per liter)

No.	Kind of Soap	1 Wash 160° F.	2 Washes 160° F.	Moisture	Weight Sample
1 a	Committee Sample, Flake Soap	64	67	1.83%	5.094
1 b	Committee Sample, Flake Soap	60–64	60–64	1.83	10.188
2	Soap Powder	60–64	67	38.4	8.851
3	Bar Soap, Silicate Filled.....	64	67	38.3	8.104
4	K—Coconut	64	64	78.0	22.50
5	K—Castor	60	64	48.5	9.709
6	K—Corn	64	64–67	48.0	9.615
7	K—Linseed	55	55–60	48.7	9.766
8	K—Oleate	55	55–60	50.6	10.012

TABLE III

1927 A. O. C. S. DETERGENCY TEST

OPTION 3

Effect of Alkaline Salts on Detergency of Commercial Flake Soap, Committee Sample

(Concentration of detergent solutions, 5 grams per liter, anhydrous basis)

A. Soda Ash Series

	Long & Hauck* 160° F.		Hoyt 160° F.		Sheely 160° F.		100° F.	
	1st Wash	2d Wash	1st Wash	2d Wash	1st Wash	2d Wash	1st Wash	2d Wash
Blank	64	64	64	67	64	64	60	67
2.5%	60–64	60–64	64	67	64	67	60	64
5%	60	60	60–64	64–67	60	64	55	60
10%	60	64–67	67	67–71	64	64	60	64
20%	55	60	64	67–71	67	71	60	64

B. Silicate of Soda ("N" silicate, 29% SiO_2, 9% Na_2O used)

	Long & Hauck* 160° F.		Hoyt 160° F.		Sheely 160° F.		100° F.	
	1st Wash	2d Wash	1st Wash	2d Wash	1st Wash	2d Wash	1st Wash	2d Wash
Blank	64	64	64–67	67	64	64	60	67
2.5%	55	55	64	64	60	64	60	67
5%	60	55	64	64	64	67	64	67
10%	64	60	64	67	67	67	60	64
20%	60	55–60	64	67	64	64	60	64

* Colors matched at a distance of 15 ft. looking across room.

Option 2 and 3. Results of collaborative work are tabulated in Tables II and III.

IV—WASHINGTON MEETING, APRIL, 1927, and COMMITTEE'S RECOMMENDATIONS

Ten members of the committee met on Saturday, April 9 at the Bureau of Standards and discussed in detail the work on the Detergency Test to date.

(a) Color Measurement. It was agreed that the present Munsell Scale left much to be desired. While the members credited Dr. Rhodes with making known to the committee an instrument, i.e. the reflectometer, of satisfactory precision for recording color values it was the consensus of opinion that the committee should concentrate its efforts on a study of the rotating disc method which seems to offer a suitable scheme for color measurement at a very moderate cost within the reach of all.

(b) Washing Machine. All of the members present united in the opinion that the present laboratory washing machine had shown itself to be entirely unsuitable for testing detergents and that it should be abandoned in favor of some small machine built to operate on the same principles as a commercial wash wheel.

(c) Methods of Soiling. It was the consensus of opinion that intensive work should be done on the problem of soiling. The present method gives a soil which is too dark and the solvent used, CCL, is not satisfactory. A variety of ideas as to soiling were advanced

(*Continued on page* 204)

Report of the Color Committee

By H. P. TREVITHICK, *Chairman*

THIS year the Color Committee has no definite recommendations to make, but can report progress. In October 1924, Mr. Bailey sent to Washington a set of Color Glasses to be standardized by the Bureau of Standards. Although we did not expect that it would take more than a year, at the last meeting of the Society in 1926 there were no indications of when the glasses would be received; nor even indications that they were being standardized. Mr. Putland was delegated to see if he could get any action. A meeting was arranged in December at the Bureau of Standards, at which Mr. Putland, Dr. Wesson and your Chairman were to be present; but due to illness in his family, Mr. Priest postponed the meeting. However, Dr. Wesson was in Washington and went out to the Bureau. Later Mr. Putland and the Chairman went to Washington, and spent a day at the Bureau, discussing the matter with Mr. Priest. At that time it was thought that the glasses would be finished in a few weeks. Now the Bureau promises them in May. In any case, it seems that we will have them before the next season begins.

Mr. Morrison of Procter & Gamble has available a considerable number of color instruments, and has made an investigation as to the best method of using these standard glasses after they have been received. He has not reported fully as yet, but believes that the usual tintometer with the split-field eye piece is as good a method of comparing glasses as any.

The Bureau of Chemistry of the Exchange has a 7.6 Red glass which was purchased from Eimer & Amend about 1912 by Charles O. Lowe. In purchasing this glass, Mr. Lowe obtained the complete stock which Eimer & Amend had at the time, some twenty or thirty glasses, and after discarding all that were obviously out of line he picked two or three that agreed and were as near the center of the lot as possible. One of these glasses was standardized by the Bureau of Standards about six years ago, and found to be 7.63 Red. This glass has been in almost constant use ever since purchased, and no particular care has been given to it. At Mr. Priest's request, this glass was submitted to the Bureau of Standards again in February and tested. The de-

H. P. Trevithick

tails of the analysis are as follows:

The Bureau's 7.6R glass (B. S. 9940) has been very carefully compared with a glass submitted by Mr. Trevithick of the New York Produce Exchange. This comparison has included:

(1) An accurate determination of the spectral transmission of each glass.

(2) A direct comparison of the glasses, each in combination with 35Y, in the Martens photometer comprising many observations by four different observers for the purpose of detecting any vanishingly small difference. It was found that when the brightness was matched, there was no certain difference in either hue or saturation.

(3) An attempt to determine experimentally any small difference in dominant wave length between these glasses when each is combined with 35Y. This proved to be too small to determine by direct observation of dominant wave lengths. The mean of 40 separate measurements by 3 different observers was found to be 0.025 millimicrons which is really less than the uncertainty of measurement.

(4) The values of dominant wave length, purity, and sunlight transmission, on the O. S. A. Colorimetry Committee basis have been computed from the data on special transmission for each of these glasses in combination with a 35-yellow glass. The computed dominant wave lengths differ by 0.02 $\mu\mu$, but the difference is contrary to that found by observation (under 3 above). The dominant wave lengths, therefore, may be considered sensibly equal. The computed purities are 0.954 and 0.953; and may be taken as sensibly equal. The computed transmissions differ by 2.2 per cent, that for the Produce Exchange glass being the less. This ratio of transmissions has since been determined directly (March 4th) on the Martens photometer and the difference found to be 2.8 per cent. It may be concluded that the transmission of the Produce Exchange glass in combination with 35y is 2.5 per cent less than the transmission of B. S. 9940 in combination with the same 35y.

The most interesting part of this test to us is that it seems to show that this particular glass at least has not changed materially since it was purchased about 15 years ago, and that therefore it seems a fair conclusion that the Lovibond glasses are pretty permanent.

The only recommendation that the Committee can make is that the Society decide definitely where the standard set is to be deposited and the charges to be made to members and non-members of the Society for standardizing the glasses.

Due to the unsatisfactory glasses which we have obtained from Lovibond, Mr. Morrison has been investigating the possibilities of having them made in this country, and he will probably report on that direct to the Society.

Mr. Priest has done a tremendous amount of work on the Lovibond glasses and on color in general and has devised new instruments and modified others to be used in this work. The Society owes him a vote of thanks for his work, as well as the gratitude of every member.

H. P. TREVITHICK.

Report of the Moisture Committee

By W. H. IRWIN, *Chairman*

FOLLOWING the untimely death of P. S. Tilson, the former chairman of the Moisture Committee, the new chairman decided to confine the co-operative work this year to a small committee and to make a study of the Bidwell-Sterling and Kingman distillation methods, and also collect data on the same samples from a number of laboratories which were equipped with a jacketed glycerin oven, similar to the one recommended by Dr. W. D. Richardson when he was chairman of the Moisture Committee in 1920.

Six samples of cottonseed meal were secured from various parts of the country, carefully prepared, and sent out at intervals to the several collaborators.

The only change made in the Bidwell-Sterling Method as sent out the previous year, was in the size of the sample (40 grams), and an instruction to the analyst to carry the distillation forward for three hours, since it had been our experience that it was necessary to carry the distillation on for this length of time to get over the last traces of water.

The Kingman Method used was the same in principle and apparatus as the Kingman Method, published in the Journal of Industrial and Engineering Chemistry, Volume 18, No. 12, December, 1926, by the Committee on Analysis of Commercial Fats and Oils of the American Chemical Society and the American Oil Chemists' Society, in the "Standard Methods for the Sampling and Analysis of Commercial Fats and Oils," the only modification being the size of the sample to be used (40 grams).

Early in the study of the methods, most of the members of the Committee reached the conclusion that the Bidwell-Sterling Distillation Method was not a satisfactory method, and that the Kingman Distillation Method, which required only about forty-five minutes as against three to four hours by the Bidwell-Sterling Distillation Method, offered more hope of a successful distillation method.

The compilation of results given below shows the results obtained by the several members of the Committee, working independently on the two distillation methods.

A. O. S. C. CO-OPERATIVE CHECK MEAL RESULTS, 1926-27

	Kingman Distillation Method					
	1	2	3	4	5	6
C. P. Long, Globe Soap Company	8.19	...	8.55	8.83	8.78	8.98
M. L. Sheeley, Armour Soap Works	8.55	9.14	7.99	8.50	8.26	7.99
C. H. Cox, Barrow-Agee Laboratories	7.75	9.90	8.95	9.72	9.18	8.92
W. D. Hutchins, Southern Cotton Oil Co	8.04	10.00	8.10	8.98	8.50	8.38
J. J. Vollertsen, Armour & Company	8.00	9.69	8.27	8.56	8.56	9.81
E. H. Tenent, International Sugar Feed Co.	8.23	9.05	...	8.74	8.39	8.50
N. C. Hamner, Southwestern Laboratories	...	...	...	8.81	8.12	8.03
W. H. Irwin, Swift & Company	8.22	9.94	8.32	8.72	8.44	8.82
Maximum	8.55	10.00	8.95	9.72	9.18	9.81
Minimum	8.00	9.05	7.99	8.50	8.12	7.99
Average	8.12	9.26	8.36	8.86	8.53	8.68

	Bidwell-Sterling Method					
	1	2	3	4	5	6
C. P. Long, Globe Soap Company	...	8.50	7.76	...	...	...
M. L. Sheely, Armour Soap Works	7.53	9.06	7.81	7.69	7.68	7.65
C. H. Cox, Barrow-Agee Laboratories	7.13	9.12	7.33	...	...	...
W. D. Hutchins, Southern Cotton Oil Co	7.81	9.70	8.17	...	...	8.27
J. J. Vollertsen, Armour & Company	7.90	...	...	8.13	8.44	8.12
E. H. Tenent, International Sugar Feed Co.	7.88	8.66	...	8.28	8.19	...
N. C. Hamner, Southwestern Laboratories	...	...	...	...	...	...
W. H. Irwin, Swift & Company	8.09	9.76	7.94	8.13	8.13	8.14
Maximum	8.09	9.76	8.17	8.28	8.44	8.27
Minimum	7.13	8.50	7.33	7.69	7.68	7.65
Average	7.72	9.13	7.80	8.06	8.11	8.04

A study of the data on the Kingman Distillation Method shows some rather erratic results. From some special work done, it appears probable that some high results were obtained due to a continuation of the distillation period for too long a time, resulting in a destructive distillation with the production of moisture. The temperature attained at the end in the Kingman Distillation Method is between 142-144°C. If the distillation is stopped promptly at this point, uniform results are obtained. If, however, the operator carries the distillation on after reaching this temperature, high and variable figures result. A modification of the Kingman Distillation Method to govern the rate of distillation, the amount of distillate to be collected, and the time of distillation, may result in a satisfactory method.

Glycerin Oven

The six samples were forwarded to fifteen Swift & Company laboratories for a moisture determination in the standard glycerin oven. The results are tabulated below:

These results, in the opinion of the Committee, are extremely satisfactory. Figures of this kind would result in no controversies between laboratories.

The Committee asked two chemical supply houses to bid on the construction of an oven of this

A. O. C. S. Co-Operative Samples of Cottonseed Meal, Numbers 1 to 6 for Moisture Determinations, Swift & Company Laboratories, Using Swift & Co. Standard Jacketed Glycerin Oven, Temp. 101° C.

Laboratory	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
1	6.91	9.00	7.36	7.98	7.94	7.39
2	7.17	8.85	7.40	7.95	7.85	7.55
3	6.90	9.12	7.58	7.81	7.67	...
4	7.20	9.05	7.56	8.03	7.80	7.58
5	6.83	9.25	7.62	8.15	7.97	7.63
6	7.00	9.03	7.49	8.11	7.93	7.54
7	6.84	9.07	7.50	7.97	7.92	7.57
8	...	9.08	7.56	7.86	7.64	7.73
9	6.80	9.05	7.50	7.99	7.78	7.59
10	6.95	9.15	7.50	8.00	7.80	7.70
11	6.97	9.17	7.54	8.06	7.88	7.58
12	7.01	9.24	7.53	8.07	7.87	7.59
13	7.21	9.30	7.69	8.18	8.02	7.65
14	6.84	9.24	7.70	8.03	7.94	7.36
15	6.94	9.10	7.54	8.05	7.84	7.77
Maximum	7.21	9.30	7.70	8.18	8.02	7.77
Minimum	6.80	8.85	7.36	7.81	7.64	7.36
Average	6.99	9.11	7.54	8.01	7.79	7.59

kind. The figures ran, depending on the number, from $195 to $250 each, which, in view of the Committee, is excessive. These ovens can be constructed and sold at a reasonable profit for $125 each. While Swift & Company are not builders of laboratory equipment, they are willing to make these ovens in their shops for this figure, if a sufficient number of laboratories indicate their desire for such an oven.

A review of the work of the moisture committees in years past seems to indicate that while a distillation moisture method is desirable that, where large numbers of samples are to be handled, an oven is much to be preferred to any distillation method.

The Committee recommends:

1. The adoption of a jacketed glycerin oven, similar in principle and design to the one recommended by Dr. W. D. Richardson in his report (1920), and used by Swift & Company laboratories in the cooperative work during the past year.

2. That the study of the Bidwell-Sterling Distillation Method be dropped, since the time required for the determination is too long and the results unreliable on cottonseed meal.

3. That the Kingman Distillation Method be studied during the coming year with the modifications suggested in the report.

W. H. IRWIN, *Chairman*,

OIL AND FAT INDUSTRIES

STATEMENT OF THE OWNERSHIP, MANAGEMENT, CIRCULATION, ETC., REQUIRED BY THE ACT OF CONGRESS OF AUGUST 24, 1912,

Of Oil and Fat Industries, published monthly at New York, N. Y., for April 1, 1926

State of New York } ss.:
County of New York }

Before me, a Notary in and for the State and county aforesaid, personally appeared John T. Ogden, who, having been duly sworn according to law, deposes and says that he is the Editor of Oil and Fat Industries, and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management (and if a daily paper, the circulation), etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, embodied in section 411, Postal Laws and Regulations, printed on the reverse of this form, to wit:

1. That the names and addresses of the publisher, editor, managing editor, and business managers are:

Publisher, Russell Palmer, 71 Columbia St., Seattle, Wash.; Editor, John T. Ogden, 220 West 42d St., New York City; Managing Editor, none; Business Manager, Russell Palmer, 71 Columbia St., Seattle, Wash.

2. That the owner is: (If owned by a corporation, its name and address must be stated and also immediately thereunder the names and addresses of stockholders owning or holding one per cent or more of total amount of stock. If not owned by a corporation, the names and addresses of the individual owners must be given. If owned by a firm, company, or other unincorporated concern, its name and address, as well as those of each individual member, must be given.) Russell Palmer, 71 Columbia St., Seattle, Wash.

3. That the known bondholders, mortgagees, and other security holders owning or holding 1 per cent or more of total amount of bonds, mortgages, or other securities are: (If there are none, so state.) None.

4. That the two paragraphs next above, giving the names of the owners, stockholders, and security holders, if any, contain not only the list of stockholders and security holders as they appear upon the books of the company but also, in cases where the stockholder or security holder appears upon the books of the company as trustee or in any other fiduciary relation, the name of the person or corporation for whom such trustee is acting, is given; also that the said two paragraphs contain statements embracing affiant's full knowledge and belief as to the circumstances and conditions under which stockholders and security holders who do not appear upon the books of the company as trustees, hold stock and securities in a capacity other than that of a bona fide owner; and this affiant has no reason to believe that any other person, association, or corporation has any interest direct or indirect in the said stock, bonds, or other securities than as so stated by him.

JOHN T. OGDEN,
Editor.

Sworn to and subscribed before me this 7th day of March, 1927.

(Seal.) Johanna E. Schult,
Notary Public Queens County No. 2766
Cert. filed in N. Y. Co. No. 1526, Reg. No. 8097a.
My commission expires March 30, 1928.

John P. Harris Joins Industrial Chemical Company

THE Industrial Chemical Company, 200 Fifth Avenue, New York City, pioneer manufacturers in this country of "bleaching" carbons, announces that John P. Harris, Chemical Engineer, who is so well and favorably known to the Edible Oil Industry as an operator, designer and builder of plants, as well as for his research work and the articles which he has published in this

John P. Harris

field in the Cotton Oil Press, National Provisioner, Chemical and Metallurgical Engineering Journal, etc., will take charge of their Chicago Office at 400 North Michigan Avenue, Wrigley Building, from which point he will cover both the Western and Southern Refining fields.

Mr. Harris will be prepared to actively service NUCHAR to the entire Edible Oil Industry and to demonstrate its high bleach ability, low oil retention, free filtration, and the removal of deleterious substances by absorption.

John P. Harris graduated from the University of Kansas in 1907, and went to work in Armour & Company's Kansas City Refinery that summer, where he continued until he went to Germany in 1908. There he passed the "Verbands Examen" at the Royal Prussian Polytechnic Institute, and worked for the Kohlbaum Company, at Charlottenburg, in the preparation of lard substitutes and margarine during 1908 and '09.

Subsequently he was for eight years in charge of Armour & Company's refineries at Kansas City and Fort Worth, and for three years Chief Chemist and Manager for the Phoenix Cotton Oil Company, at Memphis, Tenn.

He served with distinction in the Chemical Warfare Service during the World War and still holds the rank of Captain in the Chemical Warfare Service Reserve.

For two years, he was Director of Practical Research for The Institute of American Meat Packers, and has been a chemical engineer to the Edible Oil Industry practically continuously since 1918, during which time he has designed, installed and started operating some of the world's most successful refining, deodorizing and shortening plants, salad oil plants, hydrogenation plants, etc.

His developments in the field of decolorizing, filtration, clarification, etc., will be awaited with keen interest by the Industry.

Mr. Harris expects to be on hand at the A. O. C. S. and Interstate conventions at Memphis this month to greet his many friends in the Cottonseed Oil Industry and to help them solve their oil bleaching problems economically.

Report of the Referee Board of the Season 1926-1927

At the New Orleans meeting, 1926, certain recommendations were made by the Board for the improvement of the rules governing the appointment of Referee Chemists. It seemed to the members of the Board that a man doing control work for mills or refineries under contract, held practically the same position as a regularly employed chemist on the premises and his analysis in referee cases should therefore carry no added weight unless agreed to by consent of buyer and seller.

The recommendation was strenuously opposed by some of the commercial chemists present, and it was voted down. In view of the fact that various regulations have come up from time to time, it was decided wise by the Referee Board to codify rules and regulations, and recommend such improvements as seemed obvious.

Considerable correspondence was involved, and a proposed code of rules governing referee chemists was drawn up and submitted to Counsel General Benet, of the Interstate Cottonseed Crushers Association for advice and also for action by that Association. This was done as it has been the opinion of your Board for some time that many of the questions involved from time to time are intimately connected with the trading matters of the Interstate Association, and that the Referee Examining Board should be governed only by passing on the professional attainments of referees, leaving such questions as were of direct interest to the members of the Interstate to be passed upon by the Rules Committee of that Association.

In the absence of advices from Counsel General Benet, the Committee is receiving applications in the same manner as heretofore, and will consider them and other pending applications at the New Orleans meeting.

A list of Referee Chemists certified for the season 1926-1927 is given below:

REFEREE LABORATORIES AND CHEMISTS
1926-1927

Barrow-Agee Laboratories, Memphis, Tenn.; Jackson, Miss.; Shreveport, La.; Little Rock, Ark.
The Battle Laboratory, Montgomery, Ala.
Bureau of Chemistry, New York Produce Exchange, New York.
Curtis & Tompkins, San Francisco.
Forth Worth Laboratories, Fort Worth, Texas.
L. B. Forbes, Memphis, Tenn.
Geo. W. Gooch Laboratory, Los Angeles, Cal.
John C. P. Helm Laboratory, New Orleans.
Houston Laboratories, Houston, Tex.
Indiana Laboratories, Hammond, Ind.
Lehman Johnson Laboratory, Memphis, Tenn.
N. E. Katz, Meridian, Miss.
Law & Co., Atlanta, Ga.; Wilmington, N. C.
I. F. Laucks, Inc., Seattle, Wash.
Sebastian Lomanitz, Oklahoma City, Okla.
Landon C. Moore, Dallas, Tex.
Norfolk Testing Laboratories, Norfolk, Va.
Felix Paquin Laboratory, Houston, Tex.
Picard Laboratories, Birmingham, Ala.
Chas. W. Rice & Co., Columbia, S. C.
Wiley & Co., Baltimore.
Edward S. Williams Laboratory, New Orleans.

David Wesson, *Chairman.*

Report of Ammonia Committee

(*Continued from page* 186)

all results which have been received up to Wednesday morning of each week before the final proof copy is sent to the printer; have been accepted. These last results, however, have not been included in calculating the accepted average, and on one or two occasions had these results been included in the accepted average, the latter might have been changed as much as .01 per cent. This explains why some collaborators have found on one or two occasions that the accepted average as reported differed from their calculations.

This year's report of the Smalley Foundation shows much progress. More collaborators were enrolled than in any previous year while the efficiency of the analysts is improving. The value of this work is unquestionable and is being more fully appreciated each year.

The Ammonia Committee has requested no additional work from the collaborators this past year, and the foregoing summarizes the year's activity.

In concluding, the Chairman again wishes to thank the collaborators and the members of the Ammonia Committee for their cooperation.

H. C. MOORE, *Chairman*
C. A. BUTT
L. B. FORBES
H. B. BATTLE
E. H. TENENT

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Report of Detergents Committee

(*Continued from page* 196)

and those present believed that all should be tried in an effort to find the best method. Soiling by means of an emulsion of lanolin, white mineral oil and deflocculated graphite with water as the continuous phase was considered especially worthy of trial.

(d) Washing Procedure. Modification of the present method of washing was urged as regards concentration, number and duration of rinses, and elimination of heat in final preparation of the washed cloth for inspection.

V—CONCLUSION

It was clearly the consensus of opinion at the Washington meeting on April 9, 1927 that the work to date had shown the present method of evaluating detergents to be of little value. There was at the same time optimism that modification of the technique of this method along the lines outlined above might be expected to produce a workable method.

The Chairman wishes to express his sincere appreciation of the time and thought devoted to the work by members of the Detergents Committee and recommends that the work of the committee be continued along the lines indicated at the recent Washington meeting of the committee.

L. F. HOYT, *Chairman*